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NEW ORLEANS AREA WATER SUPPLY STUDY

ENVIRONMENTAL PROTECTION AGENCY

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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION VI
Dallas, Texas 75201

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16. ABSTRACT

Detailed descriptions of sampling and analytical methods used to identify and quantify trace organic compounds present in the finished (i.e. treated) water of the New Orleans, Louisiana area water supply, specifically the Carrollton Water Plant (City of New Orleans), Jefferson Parish No. 1 Water Plant (Metairie) and the Jefferson Parish No. 2 Water Plant (Marrero).

Organic compound identifications and concentrations are presented in Table 2.

Methods of analysis of carbon and resin extracts are present-d in the Appendix.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
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Submitted by

LOWER MISSISSIPPI RIVER BRANCH
SURVEILLANCE AND ANALYSIS DIVISION
REGION VI
U.S. ENVIRONMENTAL PROTECTION AGENCY
DALLAS, TEXAS

JUNE 1975

With Technical Assistance as Noted

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ACKNOWLEDGEMENTS

This project was assigned by the Region VI Administrator, U.S. Environmental Protection Agency, Dallas, Texas, to the Lower Mississippi River Branch Laboratory, Slidell, Louisiana, a facility of the Region VI Surveillance and Analysis Division. The following individuals were intimately involved in various phases of the project:

LMR Branch Chief:	Thomas F. Beckers
Sampling Coordinator:	Ernest Douglas
Analytical Coordinator and Report Preparation:	William D. Langley
Sampling Team:	Burton J. Pritchard Albert L. Hebert
Mega-Carbon Sample Processing:	Luther L. Hunt
Clerical Assistance:	Addye P. Smith

The objectives of this project could not have been accomplished without the extremely close cooperation, diversified abilities, and high competence of other individuals and groups within the Environmental Protection Agency who took time away from other duties to devote themselves to this effort.

The Analytical Chemistry Branch, Southeast Environmental Research Laboratory, Athens, Georgia, performed extensive analytical work on the Mega-carbon, CAM carbon, and Mini-carbon chloroform extracts as well as the XAD resin extracts and tetralin extracts from all three water plants sampled. Their technical assistance; timely, exceptionally thorough, and precise, is gratefully acknowledged by the Lower Mississippi River Branch personnel. Individuals at SERL involved in this project were:

ACB Branch Chief:	William T. Donaldson
Analytical:	Arthur W. Garrison Lawrence H. Keith Mike H. Carter Frank R. Allen Terry L. Floyd John D. Pope Alfred D. Thruston, Jr.

The technical assistance and close cooperation, freely given, of the Water Supply Research Laboratory, Cincinnati, Ohio, is likewise gratefully acknowledged. Their application of the relatively new volatile organics analysis technique to samples obtained from the Carrollton Water Plant enabled the detection and quantitation of certain components which were not, nor could they have been, detected by the carbon-chloroform extraction procedures. Individuals at WSRL directly involved in one or more phases of this project were:

WSRL Director:	Gordon Robeck
Analytical:	Robert Melton Frederick Kopfler E. Coleman R. Lingg Ron Dressman Don Mitchell W. Kaylor

Not the least of importance in the successful accomplishment of the analytical objectives was the job of processing the many samples obtained by the carbon adsorption methods. This must be accomplished in a well protected environment under closely controlled conditions to minimize the possibility of contamination. For the CAM and Mini-carbon samples, this job was performed by individuals within the Subsurface Environment Branch, Robert S. Kerr Environmental Research Center, Ada, Oklahoma, and the Houston Branch Laboratory of Region VI, S & A Division. These individuals were:

RSKERC, CAM-Carbon Processing:	William Dunlap Roger Cosby
Houston Branch, Region VI Mini-Carbon Processing	Medardo (Mike) Garza

Assistance in transporting the exposed CAM samples to Ada was provided by Jim Millsap of the Region VI, S & A Division, Ada Branch Laboratory.

Valuable assistance in the conduct of this project was also given by certain individuals or groups outside the Agency, namely, Mr. Gregor Junk and his associates, USAEC-Ames Laboratory, Iowa State University, Ames, Iowa, and personnel of the Gulf South Research Institute, New Orleans, Louisiana.

Finally, the entire staff of the Lower Mississippi River Branch Laboratory wish to express their sincere appreciation for the assistance and cooperation in the sampling operation extended by all plant personnel at the Carrollton Water Plant (City of New Orleans), the Jefferson No. 1 Water Plant (Metairie) and the Jefferson No. 2 Water Plant (Marrero).

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INTRODUCTION

In July, 1974, representatives of the State of Louisiana and the City of New Orleans tendered a request to the Region VI Administrator, United States Environmental Protection Agency (EPA), that a sampling and analytical survey be undertaken by the agency to determine, to the extent possible, the identities and quantitative concentrations of trace organic compounds which might be present in the finished (i.e., treated) water of the Carrollton Water Plant (City of New Orleans), Jefferson Parish No. 1 Water Plant (Metairie), and the Jefferson Parish No. 2 Water Plant (Marrero). The request was accepted and agreed to by the Regional Administrator. Immediately thereafter, a plan and schedule were formulated for conducting the necessary sampling, and arrangements were made for technical assistance in processing and analyzing the samples obtained. The assignment for sampling and analytical coordination was given to the Lower Mississippi River Branch of the Region VI Surveillance and Analysis Division. This branch facility was instructed to have sampling operations completed by mid-August, 1974, and an analytical report issued by the end of October. On November 8, 1974, the Draft Analytical Report was released pursuant to these instructions. While this report listed 66 organic compounds which had been identified and in most cases quantitated in one or more of the tested waters, it was stated that the report was not to be considered final as there were some samples remaining to be analyzed and that this analytical work was proceeding. All analytical work has now been completed, and the present report can be considered the final report for this project.

Subsequent to the release of the Draft Analytical Report by Region VI, the National Administrator of the Environmental Protection Agency directed that a nationwide survey for trace organics in drinking water supplies be initiated, a study which is currently in progress at the Federal level. It is, therefore, emphasized that this report is not a part of that nationwide survey but is solely related to the New Orleans area study previously committed. It is also emphasized that the study reported herein was strictly an analytical survey and did not encompass an evaluation of the public health significance of the compounds identified.

In accordance with directions from the Regional Administrator, a comprehensive sampling and analytical program was developed and placed in operation. This program is illustrated in Table 1 and described in some detail in the Summary of Experimental Methods. The carbon adsorption methods sampling program was established on the assumption that an individual might consume one liter of water per day, i.e., approximately one quart. Thus the use in Table 1 and elsewhere throughout this report of the terms 70-year equivalent, 10 year equivalent, etc. has reference to the volume of water sampled equivalent to the amount a person might consume in that period of time at the one liter per day rate. The other methods were added to the project either by specific request or to provide a means of detecting compounds of a type not amenable to detection by the carbon adsorption-chloroform extraction methods. In some cases it was hoped to provide some comparative evaluation of sampling methods in an as yet experimental stage of development.

To perform the necessary analtyical work for this project, the analytical coordinator through the Regional Administrator requested and was granted the technical assistance of several groups within the Environmental Protection Agency's Research Centers having highly developed and competent analytical expertise and the necessary instrumentation to perform the required analytical operations. While their assistance has been acknowledged in a previous section, it should be stressed that the results presented herein represent the combined analytical efforts of personnel of the Analytical Chemistry Branch, Southeast Environmental Research Laboratory (EPA), Athens, Georgia, and the Water Supply Research Laboratory, EPA, Cincinnati, Ohio.

SUMMARY OF EXPERIMENTAL METHODS

Shown in Table 1 is a distribution of work operations for the New Orleans area water supply study. This table provides in short and concise form information on the various sampling methods employed, the specific water plants sampled, the inclusive dates on which sampling was performed, the group performing the sampling operation, the water volume sampled, the group preparing the sample for analysis, and finally the group performing the analyses. Given below is a brief narrative description of the various sampling, sample preparation, and analytical methods used.

Sampling Methods

Carbon Adsorption. Three types of carbon adsorption units were used in the sampling performed on this project.

(1) The Mega sampler is a trailer mounted unit fabricated by personnel of the Region VI Lower Mississippi River Branch Laboratory for a previous project (ca. 1968-70). It was retrieved for purposes of the present project from NERC-Cincinnati. The Mega Sampler consists of four 12" X 30" cylindrical PVC columns which can be packed with activated carbon and connected in series. It is also equipped with two Universal Pool Products sand pre-filters for use when turbidity or sediment must be removed. For this project, however, the sand filters were unnecessary and were therefore not used. In sampling the finished water at the Carrollton Water Plant, the only location where the Mega sampler was used, two of the carbon units were employed; the remaining two were bypassed. Each of the two columns was packed with activated carbon in the following manner: the lower strata was a 5" layer of coarse

grade carbon (Pittsburgh Carbon Company Type F-300); the middle strata was a 12" layer of fine grade carbon (Nuchar C 190 X 30 mesh); and the top strata was another 5" layer of the coarse grade carbon. The total weight of carbon in each unit was approximately 22 pounds.

(2) The CAM samplers used in this project were similar to the system described in Standard Methods for The Examination Of Water And Wastewater, 13th Edition (1971) Method 139B, p. 264. The carbon unit is a 3" X 18" Pyrex cylinder which was packed with approximately 12 ounces of Nuchar C 190 X 30 mesh. In collecting the 70-year equivalent samples, two packed columns connected in series were used at each plant. In collecting the 10-year and 1-year equivalent samples, only one unit per sample was used.

(3) The Mini-sampler is a miniaturized version of the CAM sampler. The carbon column is of PVC construction and of sufficient capacity to contain 70 grams of 14 X 40 mesh activated carbon. As is the CAM sampler, this unit is equipped with flow measuring and control devices. The Mini-sampler unit is completely described in the publication The Determination Of Organics-Carbon Adsorbable In Water, 3rd Edition, by J. K. Carswell, R. W. Buelow, and J. M. Symons, U.S. EPA Water Supply Research Laboratory (1972). The actual units used in this project were obtained on loan from WSRL-Cincinnati.

In all of the carbon adsorption sampling operations described above and in Table 1, the EPA personnel from the Lower Mississippi River Branch were assisted by local operating personnel of the respective water plants.

XAD Resin Adsorption. This sampling method, developed by the USAEC-Ames Laboratory, Iowa, uses 5 cc of a macroreticular synthetic resin (Rohm and Haas XAD-2) contained in a small glass column. Its dimensions allow it to be quickly connected to any standard plumbing fixture using non-contaminating connections. The unit consists solely of the extraction column and is not equipped with flow control or automatic measuring devices. Flow rate is obtained by collecting a measured volume in a known period of time. The water sampling operations with the XAD resin adsorption units were performed on this project by Mr. Junk of the USAEC-Ames Laboratory assisted by EPA personnel. For reference to the XAD resin adsorption method see Junk, G. A., et al., "Use of Macro-reticular Resins in the Analysis of Water for Trace Organic Contaminants" J. of Chromatog, 99, p. 745-762, 1974.

Liquid-Liquid Extraction. This sampling technique was adopted by general agreement among the analysts prior to the initiation of the project with the intention that it would facilitate the recovery and analysis of highly volatile organics which it was feared might be lost in the sample processing procedures associated with the adsorption techniques. At each plant, triplicate one liter samples of finished water were extracted in separatory funnels with 2 ml of high boiling solvent, tetralin. The immiscible liquid phases were allowed to separate, the water drained and discarded, and the tetralin recovered into septum vials (Teflon-lined septums), sealed and delivered to Southeast Environmental Research Laboratory for analysis. This sampling operation was performed by personnel of SERL and LMRB.

Reverse Osmosis. This semi-permeable membrane water purification method, while proven effective for certain purposes, is in an as yet experimental stage of development for use as a solute concentration method to facilitate trace organics analysis. Its application for this purpose is undergoing evaluation at the EPA Water Supply Research Laboratory which requested its inclusion in the project with sampling performed by Gulf South Research Institute, New Orleans, Louisiana. No analytical data from this technique were derived for inclusion in this report.

It should be stated, however, that reverse osmosis is being used at WSRL primarily as a method to generate samples of organics for toxicity studies and not as a concentration step for trace organic analysis.

Volatile Stripping. (Volatile Organics Analysis, VOA; Bellar Technique). This relatively direct sampling and analytical technique employs helium gas stripping of the more volatile organics from a small water sample with entrapment of organics on a Tenax or Chromosorb 103 column. This column is then attached to the injection port of a gas chromatograph or GC/MS instrument. At elevated temperature and with carrier gas flow through the column, the components are desorbed directly into the analytical instrument.

Under the direction of the Water Supply Research Laboratory, samples by this technique were collected from a finished water tap in the Carrollton Water Plant on September 23, 1975, by personnel of the Gulf South Research Institute. Several 50 ml vials specially prepared to eliminate any possible organic contamination were provided by WSRL. Samples thus collected in a carefully prescribed manner were pre-chilled in crushed ice and shipped by

air freight to WSRL in Cincinnati for analysis. Details of this procedure, developed prior to and independently of this project, are found in the publication entitled "The Determination of Volatile Organic Compounds at the µg/l in Water by Gas Chromatography" by T. A. Bellar and J. J. Lichtenberg, Journal of the American Water Works Association, December, 1974.

Sample Preparation Methods

The description of the sample preparation methods given below is devoted solely to the process operations which followed the carbon adsorption and XAD resin adsorption methods of sample collection. Samples collected by liquid-liquid extraction and for volatile stripping required no further processing prior to analysis beyond that already described. For reasons mentioned above, the reverse osmosis samples are not considered further in this report.

Mega Carbon Processing. Immediately following its use in sample collection at the Carrollton Water Plant, the Mega sampler was transported to the EPA-LMRB laboratories where the activated carbon was removed, distributed in trays in a forced draft convection oven equipped with an activated carbon air intake filter to prevent contamination from laboratory air and dried for 10 days at a temperature of 40°C. After drying, the carbon was sealed in new, pre-cleaned, five gallon cans and taken on commerical aircraft to the Robert A. Taft Center in Cincinnati. Using the large scale permanently installed extraction unit specifically fabricated for Mega-sampler carbon extraction, the carbon was extracted for 40 hours with 50 gallons of Analytical Reagent grade redistilled chloroform. Following the 40 hour reflux extraction, the extract was concentrated by conversion of the unit to a distillation mode and distilling the excess solvent until a final volume of approximatley 1/2 gallon remained in the kettle. The concentrated chloroform extract was then recovered and transported in sealed Teflon bottles to the Southeast Environmental Research Laboratory for analysis. The Mega carbon processing was performed by Mr. Luther Hunt of the LMRB laboratory staff.

CAM Carbon Processing. On removal from the sampling location, the CAM carbon cylinders were drained of excess water, sealed, and shipped by commercial air carrier to Oklahoma City where they were claimed by personnel of the Robert S. Kerr Environmental Research Center, Ada, Oklahoma, and taken to the center by private aircraft. The columns were stored at 4°C until carbon processing could be initiated.

Columns were opened in a special carbon handling room designed to minimize the potential for contamination. The carbon was transferred to Pyrex glass dishes and dried at 35-38°C for 48 hours under a gentle flow of clean air in a mechanical convection oven. The oven air inlet was equipped with a carbon filter to prevent atmospheric contamination.

The dried carbon was transferred to 2200 ml soxhlet extractors and extracted for 48 hours with chloroform. The chloroform extracts were filtered through solvent extracted glass fiber filters to remove carbon fines and then vacuum concentrated at temperatures not exceeding 27°C in rotary evaporators to final volumes of 30-60 ml. The concentrated extracts were transferred quantitatively to 10 ml ampules, several ampules being required to accommodate each extract. The ampules were purged with clean, dry nitrogen and sealed while the contents were held at -50°C in a cold bath. The filled ampules were maintained under regrigeration (4°C) until shipment by air mail to the Southeast Environmental Research Laboratory for analysis. CAM carbon processing was performed by Mr. Roger Cosby under the direction of Dr. William Dunlap at the Robert S. Kerr Center.

Mini Carbon Processing. The exposed Mini-sampler columns, drained and sealed, were forwarded by air express to the EPA Region VI Houston Laboratory. There the carbon was removed and dried at 39.5°C for a period of 48 hours in a convection oven with activated carbon air intake filter. The dried carbon was transferred to Soxhlet extractors equipped with fritted glass thimbles and extracted for a period of 48 hours with spectrophotometric quality chloroform.

Each of the two-month equivalent sample extracts were split in a 1:1 proportion with one portion being evaporated to dryness at 70°C for carbon-chloroform extract (CCE) residue determination and the other portion reduced in volume in Kuderna-Danish evaporative concentrators. The reduced volume concentrates were quantitatively transferred to 25 ml volumetric flasks and made up to volume with chloroform. The one-day equivalent sample extracts were not split for residue determination, but the concentrative evaporation procedures were followed.

The 25 ml extracts were later transferred into vials, sealed, and shipped to the Southeast Environmental Research Laboratory for analysis. The Mini-carbon processing was performed by Mr. Medardo Garza of the EPA Houston Laboratory.

XAD Resin Processing. The XAD resin units, after exposure, were hand carried or sent by air carrier to Mr. Gregor Junk at Ames, Iowa. There the samples were extracted with redistilled ethyl ether by Mr. Junk or his associates in accordance with his procedures. The ether was dried and concentrated to 1 ml in a micro Kuderna-Danish evaporator for GC and MS analysis at Ames. One fourth of each extract was carried to Southeast Environmental Research Laboratory for analysis.

Analytical Methods

With the exception of some analyses on resin extracts performed at Mr. Gregor Junk's laboratory, all analyses performed for this project were by the Analytical Chemistry Branch of the Southeast Environmental Research Laboratory, Athens, Georgia, and the Water Supply Research Laboratory in Cincinnati. The methods described below are intended only as a brief summary of the predominance of analytical work on which the reported results are based. A more complete description of the work performed at SERL is given in the Appendix. The work performed at WSRL has been further developed in the course of the ongoing nationwide drinking water survey and will undoubtedly be reported in detail in future publications of that group.

Southeast Environmental Research Laboratory

GC and GC/MS (Gas Chromatography-Mass Spectrometry)

Gas chromatography was performed using a Varian 1400 GC with a flame ionization detector. Typical GC conditions were:

Column:	10' x 1/8" i.d. glass
Packing:	3% SP-2100 on 80/100 mesh Supelcon AW-DMCS
Program:	6 min. initial hold; then from 40 - 280°C at 6°/min.
Carrier Gas:	Helium at 20 ml/min.
Sample Size:	2 microliters

For the tetralin extracts, the temperature program was usually a 1 minute hold at 35°C (with the oven door open) followed by an increase to 210°C at 10°/min.

GC/MS instrumentation was a Finnigan 1015 system interfaced via a Gholke separator to a modified Varian 1400 GC. This system was interfaced to a Systems Industries System 150 computer for data aquisition, data storage, data reduction and manipulation. Mass spectrometer electron voltage was 70.

Some initial GC/MS work was done on a Varian MAT CH5/DF system interfaced to a Varian 2740 GC via a Watson-Biemann separator and to a Varian SS-100 Data System. This instrument was later used for confirmation of the presence of Atrazine in the Carrollton 70 year CCE by accurate mass measurement at a resolution of about 5000 amu.

Gas chromatography on these GC/MS systems was performed using a similar column and conditions to those employed in the GC runs described above.

Mass spectra stored on disks from the Finnigan GC/MS runs were compared via acousticoupler connection with spectra in the EPA-Battelle computer files at Battelle (Columbus).

Quantitative Analysis

The Perkin-Elmer PEP-1 Data System, interfaced to a Varian 1400 GC containing a SP-2100 column and operated under the conditions described above, was used for computerized quantitation and retention time measurements. Since Atrazine was present in all extracts of New Orleans samples, it was chosen as an internal standard. A stock solution of 5 parts per thousand of Atrazine (99.7% pure) in chloroform was the reference for quantity of all identified compounds for which standards were obtained. Standards, obtained from the laboratory supply or from commercial sources,

were mixed in known concentrations with other standards and with a known amount of the Atrazine reference stock solution. Mixtures were designed so as to obtain good peak resolution. The Atrazine was assigned a flame response of 1.000 and, since its concentration was known, the computer system was able to calculate the flame response, as well as the relative retention time, of each standard.

After tentative identification of compounds by GC/MS, a PEP-1 computer program was written for the GC-computer run of each extract, allowing the computer to use the known flame responses to calculate concentrations. In some cases, the flame response calculated for a standard was also used for other compounds of the same chemical class. The relative retention times, calculated for all compounds and printed out by the computer, were then manually compared with those of the available standards. It was necessary to dose the blanks with Atrazine as an internal standard, since it was ascertained that Atrazine was not present in them.

In most cases, mass spectra of the standards was obtained on the Finnigan GC/MS system for visual comparison with those of the identified compounds.

Water Supply Research Laboratory

Volative Organics Analysis

In the various analyses performed at the Water Supply Research Laboratory, the following instruments were used:

1. Perkin-Elmer Model 900 GC
2. Finnigan 1015D - System Industries 150 GC/MS
3. Phase Separations Limited TOC Apparatus

The volatile organics were purged from aliquots of the water and adsorbed on a small column containing either Tenax or Chromosorb 103 as described by Bellar and Lichtenberg. Qualitative analysis was accomplished by GC/MS using a Chromosorb 101 column and operating the mass spectrometer in the electron impact ionization mode.

Quantitative analysis of the major components of the volatile organics was accomplished by gas chromatography using the Perkin-Elmer gas chromatograph fitted with a 6 foot column of Chromosorb 101 and flame ionization detectors.

Standards of chloroform and dichloroethane were prepared by introducing 5 µl and 2.5 µl respectively into one liter of distilled water with a 5 µl syringe. This was thoroughly shaken until dissolution was complete. This stock solution was then diluted 100 fold resulting in concentrations (calculated from literature values of the densities) of 78 µg/l chloroform and 31 µg/l dichloroethane.

All Head Gas (HG) and VOA samples were injected onto a 6' x 2 mm ID 50/60 mesh Chromosorb 101 GC column. Some confirmatory analyses were performed on a hexane:ethyl ether extract (85:15 v/v). These samples were run on a 8' x 2 mm ID 3% OV-17 GC column. TOC analyses of the water were performed with the Phase Separations Limited apparatus.

The GC/MS was calibrated according to EPA standard procedures as given by J. W. Eichelberger, L. E. Harris and W. L Budde, Anal. Chem. 45, 227 (1974).

Processing of Blanks

The foregoing discussion of sampling methods, sample preparation, and analytical procedures has been devoted to the processing of actual samples. In order to assure that components identified were actually derived from the original samples and were not artifacts, contaminants, or inherent components deriving from the sampling method itself, or from the sampling media, commercial solvents used for extraction, or from any stage of the sample preparation, it was necessary to process blank samples taken through all stages of the operations in parallel with the actual samples, except that there had been no exposure to the water from the water plant undergoing test.

An exception to the latter statement existed in the development of a blank for the Mega-carbon sampling and extraction procedure. In the case of the CAM and Mini-carbon procedures, blanks were developed by passing distilled water through packed units at the Lower Mississippi River Branch Laboratory. This carbon was thereafter dried and extracted and otherwise handled in an identical manner to that of the samples. However, the large scale of the Mega-carbon unit rendered this procedure infeasible for the development of a Mega-carbon blank. In this instance, two of the Mega-carbon adsorption cylinders were packed with carbon in the same manner as previously described for the samples and the unit was returned to the Carrollton plant. For blank development, 100 gallons of Carrollton water was passed through the two units connnected in series. Following this short-term exposure, carbon from the first unit was discarded without processing while the carbon from the second unit was

dried and extracted in the same manner as the combined carbon from the primary Mega-carbon sample. This procedure for blank development rested on the assumptions that the first unit would serve as a pre-filter for organics adsorption and that the volume of 100 gallons used in wetting the carbon was insignificant in comparison to the 300,000 gallons used in the sample development. In each instance of the extraction procedures, additional solvent blanks were developed separately and independently of the extraction blanks by taking a portion of the solvent through the concentration procedure without using it for extracting carbon.

As a consequence of this processing and analysis of blanks, in the interpretation of analytical results no components could be accepted as deriving from the finished water samples unless these components were not present at a significant level in the blanks relative to the samples or unless they were identified independently in one or more of the other methods in which they were absent from the corresponding blank.

TABLE I

DISTRIBUTION OF WORK OPERATIONS

NEW ORLEANS AREA WATER SUPPLY STUDY

Sampling Method	Method Modification	Plants Sampled	Dates Samples	Sampled By	Water Volume	Sample Prepared By	Analysis
Carbon Adsorption with Chloroform Extraction	Mega Sampler	Carrollton	7/17-24/74	LMF	300,000 Gals.	LMF	SERL
	CAM 70 yr. equiv.	Carrollton	7/18-24/74	LMF	6,750 Gals.	RSKERL	SERL
		Jefferson No. 1	7/24-8/2/74	LMF	6,759 Gals.	RSKERL	SERL
		Jefferson No. 2	7/24-8/2/74	LMF	6,707 Gals.	RSKERL	SERL
	CAM 10 yr. equiv.	Carrollton	8/6-7/74	LMF	963 Gals	RSKERL	Stored in
		Jefferson No. 1	8/6-7/74	LMF	965 Gals.	RSKERL	sealed vials
		Jefferson No. 2	8/6-8/74	LMF	1,300 Gals.	RSKERL	at RSKERL
	CAM 1 yr. equiv.	Carrollton	8/13/74	LMF	74 Gals.	RSKERL	Stored in
		Jefferson No. 1	8/13/74	LMF	90 Gals.	RSKERL	sealed vials
		Jefferson No. 2	8/13/74	LMF	97.5 Gals.	RSKERL	at RSKERL
	Mini-Sampler 2 Mo. equiv.	Carrollton	7/30-31/74	LMF	62 liters	HNF	HNF-SERL
		Jefferson No. 1	7/31-8/1/74	LMF	65 liters	HNF	HNF-SERL
		Jefferson No. 2	8/1-2/74	LMF	60 liters	HNF	HNF-SERL
		Carrollton (repeat)	8/6-8/74	LMF	58 liters	WSRL	WSRL
	Mini-Sampler	Carrollton	8/6/74	LMF	1 liter	HNF	HNF-SERL
		Jefferson No. 1	8/6/74	LMF	1 liter	HNF	HNF-SERL
		Jefferson No. 2	8/6/74	LMF	1 liter	HNF	HNF-SERL

TABLE I (CONTINUED)

DISTRIBUTION OF WORK OPERATIONS

NEW ORLEANS AREA WATER SUPPLY STUDY

Sampling Method	Method Modification	Plants Sampled	Dates Sampled	Sampled By	Water Volume	Sample Prepared By	Analysis By
XAD Resin Adsorption with Ethyl Ether Extraction	Developed by the USAEC-Ames Laboratory	Carrollton	7/30-8/1/74	LMF-Junk	318 liters	Junk-SERL	SERL
		Jefferson No. 1	7/30-31/74	LMF-Junk	365 liters	Junk-SERL	SERL
		Jefferson No. 2	7/31-8/1/74	LMF	275 liters	Junk-SERL	SERL
Liquid-Liquid Contact Extraction	Tetralin Solvent	Carrollton	7/31/74	SERL-LMF	3 ea. X 1 liter	SERL	SERL
		Jefferson No. 1	7/30/74	SERL-LMF	3 ea. X 1 liter	SERL	SERL
		Jefferson No. 2		LMF	3 ea. X 1 liter	SERL	SERL
Reverse Osmosis	Cellulose Acetate Membrane in Series with Dupont Permasep Membrane	Carrollton	8/7-9/74	GSRI	Approx. 400 Gals.	WSRL	WSRL
Volatile Stripping	Bellar Technique for Volatile Organics Analysis (VOA)	Carrollton	9/23/74	GSRI	50 ml vials	WSRL	WSRL

Key to abbreviations used in Table I

LMF: Lower Mississippi River Facility (Region VI EPA) Slidell, Louisiana
SERL: Southeast Environmental Research Laboratory (EPA; NERC-Corvallis) Athens, Georgia
RSKERL: Robert S. Kerr Environmental Research Laboratory (EPA; NERC-Corvallis) Ada, Oklahoma
WSRL: Water Supply Research Laboratory (EPA; NERC-Cincinnati) Cincinnati, Ohio
GSRI: Gulf South Research Institute, New Orleans, Louisiana
HNF: Houston Facility (Region VI, EPA) Houston, Texas

ANALYTICAL RESULTS

The trace organic compounds or organic isomers of undetermined specific structure which were identified by one or more methods in samples derived from the finished water at the Carrollton Water Plant, Jefferson Parish No. 1 Water Plant, and Jefferson Parish No. 2 Water Plant are listed in Table 2. Supporting data for these identifications exist at the Water Supply Research Laboratory (Cincinnati, Ohio) or at the Southeast Environmental Research Laboratory (Athens, Georgia). Compounds are listed in Table 2 in the alphabetical order of their capitalized letter. Each compound is numbered in the order of its listing. Any reference to a compound in this discussion will be by its assigned number except where use of the name may be necessary or desirable to clarify the selected nomenclature.

No effort was made to employ a consistent nomenclature system in this list of compounds. The name selected for a specific compound may be the name most commonly used for it or by which it might be most readily recognized. For example, compound 2 is called acetone although it might also be correctly named dimethyl ketone or propanone. In some instances the common name by which a compound is preferentially listed is followed in parentheses or brackets by the name of the compound in the IUPAC (International Union of Pure and Applied Chemistry) nomenclature system. This system provides detailed information on molecular structure not given by most common chemical names. Where this style of listing was employed, it was done primarily to enable the reader to discern similarities or slight differences in certain compounds appearing in the list, as for example, compounds 3 and 14 or compounds 70 and 71.

It is not intended that the name given for compound 4 (Alachlor, one chlorine homolog) be taken as a legitimate compound name. Rather, it is a designation used for an identified compound for which a satisfactory common name was not found but which was determined by mass spectral evidence to have a homologous relationship to Alachlor, compound 3. It was determined that compound 4 was identical to compound 3 except for the inclusion of an additional chlorine atom probably bonded to the same carbon atom as the single chlorine atom in compound 3. However, due to the lack of an available standard of compound 4 for absolute confirmation of the identification, use of the probable IUPAC name for compound 4 could not be justified.

Likewise, a close relationship exists between compound 8 (Atrazine) and compound 9 (designated as Atrazine, deethyl). Compound 8 contains an ethylamino group bonded to the fourth atom of the six membered triazine ring. In compound 9 this position contains simply an amino group. The designation for compound 9 is thus intended to express its similarity to Atrazine with the absence of the ethyl group. In this instance, however, inclusion of the IUPAC name was justified as a standard was obtained for confirmation.

Compound 82, now named as 1, 3, 5-trimethyl-2, 4, 6-trioxo-hexahydrotriazine, was originally named in the draft report as trimethyl-trioxo-hexahydrotriazine isomer. The present structure has been confirmed by comparison with a standard. The compound having the structure as presently given may also be called the trimethyl ester of isocyanuric acid or the trimethyl ester of tricarbonimide.

Where the name given for a compound is followed by the term isomer, manual or computerized interpretation of the mass spectral data did not permit the analysts to determine which one of more than one possible molecular isomers bearing that name was present. Compounds 5, 6, and 7 are designated as alkylbenzene - C_2, C_3, and C_4 isomers, respectively. There are four possible C_2 isomers of an alkylbenzene. Of these, two are located elsewhere in the list as compound 51, ethylbenzene, and compound 86, o-xylene. Compound 5 must then be m-xylene, p-xylene, or a mixture of the two. There are three alkylbenzene - C_3 isomers located elsewhere in the list. Compound 52 is o-ethyltoluene; compound 53 is m-ethyltoluene; and compound 81 is designated as trimethylbenzene isomer. Compound 6 is thus most likely p-ethyltoluene or n-propylbenzene. There are no alkylbenzene - C_4 isomers other than compound 7 located elsewhere in the list.

Given for most compounds in Table 2 is a quantitative value representing the "highest measured concentration" in micrograms per liter (parts per billion). With the exceptions of compounds 22 and 34 (which were determined by Volatile Organics Analysis directly from the water medium) all concentration values were obtained through quantitative GC/MS analyses of processed carbon-chloroform extracts or XAD resin-ethyl ether extracts related back to the water medium from which the samples were derived. This concentration relationship was possible since the volumes of water passing through the carbon or resin units were known. Therefore an expressed concentration value from any component listed in Table 2 refers to concentration of that component in the water medium.

In order to express exact concentration values in the water, however, it would be necessary to know the recovery efficiency values for every stage of the sample collection, preparation, and analytical process. That is, one would need to measure with standards for each compound the efficiency of the carbon or resin adsorption from water, component losses through volatility incurred in drying the carbon, the efficiency of desorption by solvent extraction from the carbon, and losses incurred in concentrating the solvent to a low volume for analytical processing. For this project, determination of these efficiencies could be considered an impossible, or at least infeasible, task. Consequently, it is emphasized that the concentration values reported must not be interpreted as absolute concentration levels present in the water, but simply represent the highest concentration values measured by the analysts. The term "highest" is used because when values determined by two or more different methods differed to any extent, the higher or highest of the values was reported in the tabulation. All values are retained in the analysts' records. The reader who is familiar with the Draft Analytical Report for this project, previously released, will note that values for a number of compounds are significantly higher than previously reported. This is due to the fact that higher values for these compounds were found on analysis of the 2-month mini-sample extracts which had not been performed at the time of the earlier report.

It is also recognized that all of the specific organic compounds identified and reported herein were, in the final stage of analysis,

determined through some modification of gas chromatography. For a compound to be analyzed by this technique, it must possess some degree of volatility under the conditions of analysis. Consequently, non-volatile organic substances would not have been detected under the analytical conditions employed. The compounds listed in Table 2 represent those compounds which have been identified and quantitated with a reasonable degree of certainty by the analysts from the samples presented to them for analysis.

TABLE 2

ORGANIC COMPOUND IDENTIFICATIONS
NEW ORLEANS AREA WATER SUPPLY STUDY

	Compound	Highest Measured Concentration µg/l (ppb)		
		Carrollton Water Plant	Jefferson #1 Water Plant	Jefferson #2 Water Plant
1	Acetaldehyde	D-VOA	NE	NE
2	Acetone	D-VOA	NE	NE
3	Alachlor * [2-chloro-2',6'-diethyl-N-(methoxy-methyl)acetanilide]	1.9	2.9	2.1
4	Alachlor, one chlorine homolog	1.7	P	0.05
5	Alkylbenzene - C_2 isomer (m-Xylene or p-Xylene)	7.5	5.6	6.?
6	Alkylbenzene - C_3 isomer (p-Ethyltoluene or n-Propylbenzene)	2.4	1.9	2.2
7	Alkylbenzene - C_4 isomer	< 0.1	ND	ND
8	Atrazine * (2-chloro-4-ethyl-amino-6-isopropyl-amino-s-triazine)	4.9	5.2	5.4

TABLE 2 (Continued)

ORGANIC COMPOUND IDENTIFICATIONS
NEW ORLEANS AREA WATER SUPPLY STUDY

	Compound	Highest Measured Concentration µg/l (ppb)		
		Carrollton Water Plant	Jefferson #1 Water Plant	Jefferson #2 Water Plant
9	Atrazine, deethyl* (2-chloro-4-amino-6-isopropylamino-s-triazine)	0.78	0.80	0.75
10	Benzaldehyde *	0.03	ND	ND
11	Benzyl butyl phthalate *	1.4	1.8	1.6
12	Bromodichloromethane	< 0.1	< 0.1	ND
13	Bromoform *	0.57	ND	ND
14	Butachlor * [2-chloro-2',6'-diethyl-N-(butoxymethyl) acetanilide]	0.05	0.06	0.05
15	Butanone	D-VQA	NE	NE
16	Butyl octyl maleate *	D-RE(J)	D-RE(J)	ND
17	Carbon disulfide	D-VQA	NE	NE

TABLE 2 (Continued)

ORGANIC COMPOUND IDENTIFICATIONS
NEW ORLEANS AREA WATER SUPPLY STUDY

	Compound	Highest Measured Concentration µg/l (ppb)		
		Carrollton Water Plant	Jefferson #1 Water Plant	Jefferson #2 Water Plant
18	Carbon tetrachloride	D-VOA	NE	NE
19	α-Chlordane	<0.1 (T)	ND	ND
20	Chlordene *	<0.1 (T)	ND	ND
21	bis (2-Chloroethyl) ether *	0.04	0.16	0.12
22	Chloroform *	133 (a)	NE	NE
23	bis(2-Chloroisopropyl) ether *	0.18	0.08	0.03
24	m-Chloronitrobenzene *	D-RE(J)	D-RE(J)	ND
25	Cyanazine * [2-(4-chloro-6-ethylamino-s-triazin-2-ylamino)-2-methylpropionitrile]	0.35	0.21	0.31
26	DDE * [2,2-bis(p-chlorophenyl)-1,1-dichloroethylene]	0.05(J)	0.05(J)	ND

TABLE 2 (Continued)

ORGANIC COMPOUND IDENTIFICATIONS
NEW ORLEANS AREA WATER SUPPLY STUDY

	Compound	Highest Measured Concentration µg/l (ppb)		
		Carrollton Water Plant	Jefferson #1 Water Plant	Jefferson #2 Water Plant
27	n-Decane *	2.4	2.0	2.0
28	Decane, branched isomer	5.8	5.4	5.2
29	Dibromochloromethane *	1.1	0.4	0.06
30	Dibromodichloroethane isomer	0.33	ND	0.63
31	Dibutyl phthalate *	0.10	0.36	0.23
32	2,6-Di-t-butyl-p-benzoquinone *	0.22	0.21	0.25
33	m-Dichlorobenzene *	<3	<0.1	ND
34	1,2-Dichloroethane	8 (a)	NE	NE
35	Dichloroiodomethane	1.1	1.3	1.5
36	Dichloromethane	D-VOA	NE	NE
37	Dicyclopentadiene *	ND	D-RE(J)	ND

TABLE 2 (Continued)

ORGANIC COMPOUND IDENTIFICATIONS
NEW ORLEANS AREA WATER SUPPLY STUDY

	Compound	Highest Measured Concentration µg/l (ppb)		
		Carrollton Water Plant	Jefferson #1 Water Plant	Jefferson #2 Water Plant
38	Dieldrin *	0.04	0.07	0.05
39	Diethyl phthalate *	0.24	0.10	0.18
40	Di-(2-ethylhexyl) adipate *	0.10	ND	ND
41	Di-(2-ethylhexyl) phthalate *	11	0.50	1.2
42	Dihexyl phthalate	0.05	ND	0.16
43	Dihydrocarvone	0.14	0.06	0.07
44	Diisobutyl phthalate *	0.59	ND	ND
45	Dimethyl phthalate *	0.60	0.82	0.74
46	Dipropyl phthalate *	0.07	0.13	0.14
47	n-Dodecane *	0.10	0.40	0.37
48	Endrin *	0.004 (T)	0.008 (T)	0.006 (T)

TABLE 2 (Continued)

ORGANIC COMPOUND IDENTIFICATIONS
NEW ORLEANS AREA WATER SUPPLY STUDY

	Compound	Highest Measured Concentration µg/l (ppb)		
		Carrollton Water Plant	Jefferson #1 Water Plant	Jefferson #2 Water Plant
49	Ethanol	D-VOA	NE	NE
50	Ethyl acetate	D-RE	ND	D-RE
51	Ethylbenzene	2.3	1.6	1.8
52	o-Ethyltoluene *	ND	0.04	0.02
53	m-Ethyltoluene *	ND	0.05	0.02
54	1,2,3,4,5,7,7-Heptachloro-norbornene *	0.06	0.07	0.07
55	Heptachloronorbornene isomer	0.06	0.04	0.04
56	Hexachloro-1,3-butadiene *	0.70	0.27	0.21
57	Hexachloroethane *	4.3	0.19	0.30
58	Isophorone *	2.9	2.8	9.5
59	Limonene *	0.03	ND	ND

TABLE 2 (Continued)

ORGANIC COMPOUND IDENTIFICATIONS
NEW ORLEANS AREA WATER SUPPLY STUDY

	Compound	Highest Measured Concentration µg/l (ppb)		
		Carrollton Water Plant	Jefferson #1 Water Plant	Jefferson #2 Water Plant
60	Methanol	D-VOA	NE	NE
61	Methyl benzoate *	ND	<0.01	ND
62	Methylnaphthalene *	ND	D-RE(J)	ND
63	Naphthalene *	D-RE(J)	D-RE(J)	ND
64	3-Methylbutanal	D-VOA	NE	NE
65	2-Methylpropanal	D-VOA	NE	NE
66	n-Nonane *	2.4	2.4	2.1
67	Pentachloroethane	<0.1	ND	ND
68	Pentachlorophenyl methyl ether	<0.1 (T)	ND	ND
69	n-Pentadecane *	0.03	0.10	0.10

TABLE 2 (Continued)

ORGANIC COMPOUND IDENTIFICATIONS
NEW ORLEANS AREA WATER SUPPLY STUDY

	Compound	Highest Measured Concentration µg/l (ppb)		
		Carrollton Water Plant	Jefferson #1 Water Plant	Jefferson #2 Water Plant
70	Propazine * [2-chloro-4,6-bis (isopropylamino)-s-triazine]	<0.1	< 0.1	<0.1
71	Simazine * [2-chloro-4,6-bis (ethylamino)-s-triazine]	<0.1	< 0.1	<0.1
72	1,1,1,2-Tetrachloroethane*	0.11	ND	ND
73	Tetrachloroethylene *	<1	0.20	0.20
74	n-Tetradecane *	0.10	0.10	0.12
75	Toluene *	11	7.1	12
76	1,1,2-Trichloroethane *	6.2	8.5	6.4
77	1,1,2-Trichloroethylene	D-VOA	NE	NE

TABLE 2 (Continued)

ORGANIC COMPOUND IDENTIFICATIONS
NEW ORLEANS AREA WATER SUPPLY STUDY

	Compound	Highest Measured Concentration µg/l (ppb)		
		Carrollton Water Plant	Jefferson #1 Water Plant	Jefferson #2 Water Plant
78	1,1,1-Trichloro-propane	< 0.1	ND	ND
79	1,2,3-Trichloro-propane	< 0.2	ND	ND
80	n-Tridecane *	0.30	0.17	0.20
81	Trimethylbenzene isomer	6.1	5.1	5.3
82	1,3,5-Trimethyl-2,4,6-trioxo-hexahydrotriazine *	0.01	ND	ND
83	Triphenyl phosphate *	0.12	ND	ND
84	n-Undecane *	2.5	< 10	2.1
85	Undecane, branched isomer	5.3	ND	ND
86	o-Xylene *	4.1	2.8	3.4

KEY TO SYMBOLS USED IN TABLE 2

*	While all compounds listed in Table 2 were identified by one or more methods, those marked with this symbol gained added confirmation by gas chromatography retention time match with an available standard of the compound. Compounds 38 and 48 were further confirmed by gas chromatography retention time match on two additional columns of varying polarity.
a	The quantitative values for compounds 22 and 34 were obtained on Volatile Organics Analysis by comparison with standards of known concentration at the Water Supply Research Laboratory. Compound 22 was detected but not quantified in Tetralin extracts of Carrollton water at Southeast Environmental Laboratory, but not in Tetralin extracts of Jefferson No. 1 or Jefferson No. 2, waters which were not examined by the Water Supply Laboratory. The Southeast Laboratory did not detect compound 34 in any of its processed samples.
D-VOA	These compounds were detected by Volatile Organics Analysis only as performed by the Water Supply Research Laboratory. Quantitative values were not obtained except for compounds 22 and 34 as indicated above. Only the Carrollton water was examined by this method.
NE	Not examined. This symbol is used for some compounds reported by the Water Supply Research Laboratory and not detected by the Southeast Environmental Laboratory. It is used exclusively under the headings for Jefferson No. 1 and No. 2, waters which were not examined by the Water Supply Laboratory.
ND	This symbol means the compound was not detected in a specific water by any of the methods employed. It applies mostly under the Jefferson No. 1 and No. 2 samples since the Mega-sample, obtained only at the Carrollton Plant, contains some compounds not detected by the other sampling techniques.
D-RE	This symbol means the compounds were detected, but not quantified, only in XAD resin extracts in samples analyzed at the Southeast Environmental Reserach Laboratory.
D-RE(J)	These compounds were detected but not quantified (except for DDE) in XAD resin extracts analyzed by Gregor Junk. Where the (J) symbol is used, the compounds were not detected by resin extracts analyzed at the Southeast Environmental Laboratory.

P	Present, but not quantitated.
(T)	Detected only by Thruston at the Southeast Environmental Laboratory by GC/MS after fractionation by column chromatography.

PROJECT SUMMARY AND EVALUATION

The sampling program as originally assigned to the Lower Mississippi River Branch was completed on schedule in mid-August, 1974. Sampling for volatile organics analysis, however, was performed in September by personnel of the Gulf South Research Institute under direction of the Water Supply Research Laboratory. Generally the time required in processing samples for analysis prevented getting samples to the analysts before mid-August to early September. Thus to meet the initial schedule for presentation of analytical results, the analysts had only eight to ten weeks to perform extremely complex and demanding analyses while maintaining precise control over sample integrity and adhering to scientifically defensible techniques. It is a tribute to their abilities and dedication to the established schedule that preliminary results were available for presentation in the Draft Analytical Report released by Region VI on November 8, 1974. Since that date, the analytical work for which committments were obtained has been completed and further work in the nature of confirming, adding to, or revising, to some extent, previous results have been accomplished. The present report represents the completion of this project as assigned.

The previous Draft Analytical Report listed 66 organic compounds identified with some degree of certainty in one or more of the three waters sampled. The present report lists 86 such compounds including 64 of the original 66, although some compounds previously listed as isomers of undetermined specific structure may have now been given precise names following confirmation of structure. Of the original 66

compounds listed, two were dropped from the list, these being one alkylbenzene-C_3 isomer and one undecane branched isomer. Other alkylbenzene isomers which may appear to be missing in comparison with the earlier list are among those compounds appearing elsewhere under more precise names (see Analytical Results section). In the updated list of Table 2, there are thus 22 new identifications. In addition to the 86 compounds listed, mass spectra were obtained for eight additional compounds, but the analysts were unable to interpret the spectra with sufficient certainty for precise compound identification.

There is no claim presented here, either stated or implied, that the compounds listed represent all or any known proportion of the compounds which may be present in the water at any given time. While the sampling and analytical methods used in this study are among the most versatile available, their limitations are acknowledged without apology. There are at least two relatively simple methods which are designed to provide a gross measurement of organics in water at levels which might occur in drinking water. These two methods were performed at the Water Supply Research Laboratory on Carrollton water samples or extracts during the course of this study. One of these methods is Total Organic Carbon (TOC), an instrumental measurement of elemental carbon in organic form. While several commercial instruments based on various techniques are available for accomplishing this measurement, using a Phase Separations Limited TOC apparatus, the WSRL reported to this author a value of 2.5 mg/l TOC in a water sample obtained at the

Carrollton plant on August 9, 1974. Other values obtained for TOC using an Oceanography International instrument at WSRL on 5 replicate sealed ampules of Carrollton Water (date sampled September 23, 1974) were 2.4 $\pm$ 0.2 mg/l (personal correspondence with Dr. Robert Melton, October 23, 1974). Another method for obtaining a gross measurement for organics in drinking water is the carbon-chloroform extract residue method. This method has been referenced earlier under the description of the CAM samplers in the experimental section. Using a Mini-sampler modification of this method as developed at WSRL, that laboratory obtained a value for CCE of 0.3 mg/l in a Mini-sample extract sampled 8/6-8/74 at the Carrollton plant. On the Mini-sample extract from Carrollton on 7/30-31/74, Mr. Mike Garza of the Region VI Houston laboratory obtained a value of 0.23 mg/l for CCE; for the Jefferson No. 1 Mini-sample extract of 7/31-8/1/74, he obtained a value of 0.31 mg/l; and from the Jefferson No. 2 Mini-sample extract of 8/1-2/74 he obtained a CCE value of 0.46 mg/l. The CCE residue method would reflect a gross mass measurement of organics which were relatively non-volatile, adsorbable on carbon, and extractable into chloroform. There is generally no attempt at identification of these organics.

Using the data presented in the paragraph above in comparison with the concentration data presented in Table 2, the interested reader may attempt to satisfy for himself what proportion of organics have been accounted for herein. However, recognizing the limitations of the various methods and the fact they they are only superficially related,

this author hesitates to present such an estimate for fear that such speculation would only be misleading.

Committments were not obtained for the analysis of all samples collected and processed. Those samples on which no analytical work was performed included the 10-year and 1-year equivalent CAM samples and the 1-day equivalent Mini-samples. The CAM extracts were retained under refrigeration at the Robert S. Kerr Environmental Research Center while the 1-day Mini-sample extracts are located at SERL.

The predominance of the data presented herein has been derived from the carbon adsorption sampling methods and the volatile organics analysis (VOA) technique. These two methods appear to complement each other very well. The latter technique appears to be best suited to the types of compounds that would be lost in the sample processing methods employed with carbon adsorption. Obviously, one of the major components detected by the VOA method, chloroform, would have been completely undetectable in the carbon methods where chloroform is used as a carbon extractant. The XAD resins, while not as productive of analytical data in this study, nevertheless deserve further comparison with carbon adsorption under more closely controlled conditions. The reverse osmosis methods, not a source of data in this project, are receiving further study at the research level as a potential technique of organic concentration for analytical purposes. Direct liquid-liquid extraction with tetralin was also a disappointing source of data although some compound confirmations were obtained from this source. While some comparisons of sampling and analytical

methods naturally accrued from this study, it was not a primary objective of the study to make such comparisons; therefore, it would be a mistake to make firm conclusions regarding any of the methods presented since strict sampling controls and precisely duplicated conditions were not applied.

No judgement has been presented here regarding the possible source of the identified compounds or their public health significance. This project was initiated and conducted solely as an analtyical study and the results have been presented accordingly. Questions relating to the significance of the findings are now the subject of further concern at the National level within EPA and other groups, both public and private. The current status of these ongoing studies is unknown to this author, and, in any event, would be beyond the scope of this report.

New Orleans Area Water Supply Study-
Analysis of Carbon and Resin Extracts

Prepared and Submitted to the

Lower Mississippi River Branch
Surveillance and Analysis Division
Region VI
U.S. Environmental Protection Agency

by the

Analytical Chemistry Branch
Southeast Environmental Research Laboratory
U.S. Environmental Protection Agency
Athens, Georgia 30601

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Appendix A

SUMMARY

Eighty-two organic compounds were tentatively identified by gas chromatography-mass spectrometry in one or more of the three New Orleans area finished drinking water supplies. Fifty-five of these identifications have been confirmed. Concentrations range from 0.004 µg/l to 12 µg/l.

ACKNOWLEDGEMENTS

Larry Keith, Mike Carter, Frank Allen, Terry Floyd, John Pope, Al Thruston, and Wayne Garrison of the Analytical Chemistry Branch, (ACB), SERL, spent about 20 man-months ($44,000) on this project between July 1, 1974 and April 30, 1975. The work was conducted as technical assistance to Region VI, and regularly scheduled research tasks involving the above personnel were curtailed as a result of their participation.

The administrative assistance and technical advice of the Branch Chief, William Donaldson, is appreciated. The participation of Gregor Junk, USAEC--Ames Laboratory, Iowa State University, Ames, Iowa, in the collection, extraction, and analysis of the resin adsorption samples was very helpful.

EXPERIMENTAL

SAMPLE HANDLING, EXTRACTION, AND FRACTIONATION

Descriptions of the New Orleans samples and corresponding extracts analyzed by the ACB are contained in Table 1.

Carbon Adsorption Samples

All carbon adsorption sampling was done by EPA Region VI personnel. The carbon was processed at other laboratories (Table 1); ACB personnel received only the carbon chloroform extracts (CCE's), which were refrigerated until analysis. Volumes of 70-year and mega-sample extracts were adjusted by evaporation in Kuderna-Danish evaporators so that 1 µl corresponded to 1 liter of water passed through the carbon filter. Blank extract volumes were adjusted accordingly. Mini-sample extracts were evaporated to 0.3 ml so that 1 µl corresponded to 100 ml of water. Although a number of fractionation and separation procedures were applied to the carbon chloroform extracts, as described below, most information was obtained by direct GC and GC-MS (gas chromatography-mass spectrometry) without pre-treatment.

Table 1. Description of New Orleans Drinking Water Extracts

Extract	Sample Site, Water Equivalent, and Dates Sampled			Disposition
Mega-sample CCE*	Carrollton WTP**	300,000 gal.	7/17-24/74	Extracted at AWTRL, Cinn. by Region VI personnel. Extracts were hand-carried to SERL in Teflon bottles.
Mega-sample blank CCE	----	----		
70-year filter #1, CCE	Carrollton WTP	25,500 liters	7/18-24/74	"70-year" samples correspond to the amount of water a man drinks in 70 years at 1 liter/day. Extracted by Bill Dunlap at the RSKERL in Ada, Okla., and shipped to SERL in sealed glass ampules. The #2 filters were in series with the #1 filters.
" " #2, CCE	"	"	"	
" " #1, CCE	Jefferson No. 2 WTP	"	7/24-8/2/74	
" " #2, CCE	"	"	"	
" " #1, CCE	Jefferson No. 1 WTP	"	"	
" " #2, CCE	"	"	"	
" blank CCE	----	----	----	
" filter #1, CAE***	Carrollton WTP	25,500 liters	----	
" blank CAE	----	----	----	
Mini-sample CCE	Carrollton WTP	1 liter	8/6/74	Mini-samples were extracted by Mike Garza, Region VI, Houston, and shipped to SERL in glass vials. Garza used one-half of the 60 liter extracts. The 1 liter extracts were not examined.
"	"	60 liters	7/30-31/74	
"	Jefferson No. 2 WTP	1 liter	8/6/74	
"	"	60 liters	8/1-2/74	
"	Jefferson No. 1 WTP	1 liter	8/6/74	
"	"	60 liters	7/31-8/1/74	
" blank CCE	----	----	----	
XAD Resin, ether extract	Carrollton WTP	318 liters	7/30-8/1/74	Samples were collected and resin extracted by SERL and USAEC, Ames personnel.
" " " "	Jefferson No. 2 WTP	275 liters	7/31-8/1/74	
" " " "	Jefferson No. 1 WTP	365 liters	7/30-31/74 and 7/31-8/1/74	
" " , blank extract	----	----	----	
Tetralin extract	Carrollton WTP	1 liter	7/31/74	Samples were extracted by SERL personnel in New Orleans -- see Experimental Section.
" "	Jefferson No. 2 WTP	1 liter	7/31/74	
" "	Jefferson No. 1 WTP	1 liter	7/30/74	
" " , blank	----	----	----	

*Carbon chloroform extract -- obtained by chloroform extraction of carbon through which water sample had passed.

**Water treatment plant -- samples were collected from a tap at the plant after treatment.

***Carbon alcohol extract -- obtained by extracting carbon filter #1 with ethanol after chloroform extraction.

Aliquots of the mega-sample CCE and its blank were fractionated by thin layer chromatography (TLC) to determine whether an initial fractionation could improve GC separation of components. This procedure is especially good for isolating the polynuclear aromatic class of hydrocarbons. Aliquots of the extracts were spotted on silica-gel TLC plates and developed with benzene for 10 cm. Hydrocarbons had previously been found to migrate to the 8-10 cm region of these plates, polynuclear aromatics to the 6-8 cm region, and more polar aromatic to the 0-6 cm region. These regions of adsorbent were scraped off the plates and eluted with chloroform. The eluates were evaporated to an appropriate volume for analysis by GC and GC-MS.

Aliquots of the mega-sample CCE, the Carrollton 70-year CCE, and their respective blanks were analyzed by the EPA organochlorine pesticide method.[1] This procedure involves fractionation by column chromatography with detection by electron capture GC. The presence of dieldrin and endrin, as determined by this technique, was confirmed by matching GC retention times with standards on two other GC columns of varying polarity. Further confirmation, as well as detection of chlordene, α-chlordane, and pentachlorophenyl methyl ether, was obtained by GC-MS analysis of the appropriate fractions.

The Jefferson #1 and Jefferson #2 CCE's were also analyzed by electron capture GC for the detection and measurement of endrin without prior fractionation.

Other attempts were made to improve GC peak separation by various types of fractionation. Steam distillation neatly separated the mega-sample CCE into volatile distillables and non-distillables, but did little to improve actual peak separation without major adjustment of GC conditions. Solubility extraction of the CCE into strongly and weakly acidic, basic, and neutral fractions served only to show that most of the compounds are neutral.

The carbon alcohol extract (CAE) gave only a few poorly resolved peaks when examined directly by GC. Aliquots of the extract and of the corresponding blank were evaporated just to dryness and methylated with diazomethane by a standard procedure[2], since most of the components were expected to be highly polar materials, including carboxylic acids and phenols. The methylated extracts were made to appropriate volumes and analyzed by GC and GC-MS.

Pesin Adsorption Samples

Gregor Junk, USAEC--Ames Laboratory, Iowa State University, Ames, Iowa, assisted ACB personnel in collecting resin adsorption samples in New Orleans. Rhom and Haas XAD-2 resin was cleaned and placed in glass columns according to Junk's established technique.[3] The columns were connected to finished water lines at the three New Orleans treatment plants and samples were collected for about 24 hours according to the schedule in Table 1. One Jefferson No. 1 resin sample was taken to Ames by Junk and the other samples were removed from the water lines and mailed to Junk by Region VI EPA personnel.

The resin samples and corresponding resin blank were extracted with ethyl ether according to Junk's technique as soon as they arrived in Ames. The blank consisted of an equal amount of resin of the same batch used in the sample columns. The ether extracts were dried and concentrated to 1 ml in a micro Kuderna-Danish evaporator for GC and GC-MS analysis at Ames. One-fourth of each extract was taken to SERL for GC and GC-MS analysis. Volumes were adjusted by evaporation with a stream of dry nitrogen so that 1 µl corresponded to 1 liter of water passed through the resin.

Tetralin Extractions

Triplicate 1-liter samples of finished water from each of the New Orleans treatment plants were manually extracted with tetralin (1, 2, 3, 4-tetrahydronaphthalene). The first two samples were extracted by SERL personnel in New Orleans. The Jefferson No. 2 sample was extracted by Region VI EPA personnel and mailed to SERL. A 2-ml portion of Matheson, Coleman and Bell practical grade tetralin was used to extract each sample, using the same tetralin-rinsed 1-liter separatory funnel for all samples and the blank. The tetralin had been checked by GC to assure presence of enough "window" area to observe early-eluting volatile components in the water extract. After standing for 15 minutes, the water was drained slowly from the separatory funnel, leaving 1.0 to 1.2 ml of the tetralin extract. The blank consisted of 2 ml of tetralin that had been combined with 2 ml of distilled water and used to rinse the separatory funnel prior to sample extractions. All extracts and the blank were sealed in serum vials with teflon lined septum caps. Septum caps were replaced after each syringe puncture. The volume was not adjusted prior to GC or GC-MS analyses. Extracts were stored in a refrigerator.

IDENTIFICATION METHODS

Gas chromatography was performed using a Varian 1400 GC with a flame ionization detector. Typical GC conditions were as follows:

Column: 10 ft x 1/8 in. i.d. (1/4 in. o.d.) glass
Packing: 3% SP-2100 on 80-100 mesh Supelcon AW/DMCS
Program: 6 min initial hold; then from 40°--280° at 6 °/min.
Carrier gas: helium at 20 ml/min.
Sample size: 2 µl

For the tetralin extracts, the temperature program was usually a 1 min hold at 35° (with the oven door open) followed by an increase to 210° at 10 °/min.

A Finnigan 1015 mass spectrometer interfaced via a Gohlke separator to a modified Varian 1400 GC was used for GC-MS analysis. The GC-MS was interfaced to a System Industries System 150 computer for data acquisition, data storage, and data reduction and manipulation. Ionizing voltage was 70 eV.

Some initial GC-MS work was done on a Varian MAT CH5/DF system interfaced to a Varian 2740 GC via a Watson-Biemann separator and to a Varian SS-100 Data System. This instrument was later used to confirm the presence of atrazine in the Carrollton 70-year CCE by accurate mass measurement at a resolution of about 5000. It was also used for determination of empirical formulae of the major mass spectral peaks of alachlor and the alachlor homolog.

The GC conditions for GC-MS analyses were similar to those used for GC analyses described previously.

Mass spectra stored on disks from the Finnigan GC-MS runs were compared via acousticoupler connection with spectra in the EPA-Battelle computer files at Battelle (Columbus). Mass spectra were also compared through a computer terminal and acousticoupler with spectra in the NIH Mass Spectral Search System in Bethesda, Maryland.

Chemical ionization mass spectrometry was performed on selected components in the Carrollton 70-year CCE with a separate computerized Finnigan 1015 mass spectrometer interfaced to a Finnigan 9500 GC using methane as a carrier/reactant gas.

Combination gas chromatography-infrared spectroscopy instrumentation was a computerized Digilab FTS-14D/IR Fourier transform spectrophotometer equipped with the Digilab GC/IR accessory and interfaced to a Perkin-Elmer 990 GC. This instrument was used to confirm the presence of alachlor and atrazine in the Carrollton mega-sample CCE.

QUANTITATIVE ANALYSIS

A Perkin-Elmer PEP-1 Data System, interfaced to a Varian 1400 GC operated under the conditions described above, was used for computerized quantitation and retention time measurements. Since atrazine was present in all extracts of New Orleans samples, it was chosen as an internal standard. A stock solution of 5 parts-per-thousand of atrazine (99.7% pure) in chloroform was the reference for quantitation of all identified pollutants for which standards were obtained. Solutions of known amounts of pure reference compounds were prepared and mixed with a known amount of the atrazine reference stock solution. Mixtures were designed so as to obtain good GC peak resolution. The atrazine was assigned a flame response of 1.000 and, since its concentration was known, the computer system was able to calculate the flame response, as well as the relative (to atrazine) retention time, of each standard.

After tentative identification of pollutants by GC-MS, a PEP-1 computer program was written for the GC-computer analysis of each extract, allowing the computer to use the known flame responses to calculate pollutant concentrations (Figure 1). In some cases, the flame response calculated for one standard was also used for other compounds of the same chemical class. The relative retention times, calculated for all pollutants and printed out by the computer, were then manually compared with those of the available standards. The blanks had to be dosed with atrazine as an internal standard, since atrazine was not present in them.

In most cases, mass spectra of the standards were obtained on the Finnigan GC-MS system for visual comparison with those of the identified compounds.

STANDARDS FOR CONFIRMATION

Standard reference compounds were obtained from commercial chemical suppliers (e.g., Aldrich Chemical Co. and Columbia Organics Chemical Co., Columbia, S.C.), or, in

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```
RRF     1    12: 37.0   9/  9/ 74  CCE 1 CARROLTON

INIT  9   . 3D METHOD   A2   . 3D FILE    S5 :

  TIME      AREA       RRT        RF          C        NAME
  3.48    1.4457      .107,     1.000,      .8987, !
  4.26    1.0659      .132,     1.000,      .6626, !
  5.40     .0066      .167,     1.000,      .0041, !
  6.05     .7198      .198,     1.000,      .4474, !
  6.56     .2873      .204,     1.319,      .2356, 112-TRICHLOROETHANE:
  6.89     .7221      .212,      .514,      .0775, TOLUENE:
  7.38     .4588      .228,     2.298,      .6788, CHLORODIBROMOMETHANE:
  7.99     .0368      .247,     1.000,      .0229, !
  8.42     .5856      .261,     7.500,      .9709, TETRACHLOROETHYLENE
  9.01     .0438      .279,     1.000,      .0272, !
  9.75     .0553      .302,     1.000,      .0345, !
  9.96     .0864      .308,     1.000,      .0538, !
 10.35     .1933      .320,     4.778,      .5694, BROMOFORM:
 10.64     .1883      .330,      .208,      .0502, M- OR P-XYLENE:
 11.03     .0783      .340,     1.000,      .0487, !
 11.37     .6653      .352,     1.480,      .6056, CHLOROBENZENE: P-XYLENE:
 12.02     .1120      .372,      .184,      .0128, NONANE:
 12.10    1.1663      .406,     1.000,      .7250, UNK HY 145:
 12.58     .0677      .401,     1.000,      .0421, !
 12.83     .1578      .408,      .217,      .0212, P-ET TOL OR FROM BE:
 13.04     .0664      .435,      .257,      .0398, BIS-2-CL-ET ETHER :
 13.28     .1710      .445,      .184,      .0196, BRANCHED DECANE:
 13.85     .0593      .460,      .240,      .0387, TRIMETHYL BE ISOMER:
 15.43     .3721      .480,      .184,      .0380, DECANE, UNK ARPM:
 16.15     .1760      .500,      .216,      .0346, LIMONENE:
 16.39     .5240      .508,      .557,      .1817, BIS-2-CL-IPR ETHER:
 16.69     .5580      .517,      .184,      .0538, BRANCHED UNDECANE:
 17.18    1.5324      .532,     1.635,     1.5575, HEXACHLOROETHANE:
 17.62     .0975      .546,     1.000,      .0607, !
 17.95     .0707      .556,     1.000,      .0439, !
 18.45    3.2608      .578,      .298,     1.5300, ISOPHORONE:
 20.32     .5480      .630,      .079,      .0983, DIHYDROCARVONE:
 21.16     .3021      .656,      .866,      .1629, HEXACHLOROBUTADIENE:
 21.59     .0691      .669,      .184,      .0080, N-DECANE:
 21.95     .0418      .680,     1.000,      .0260, !
 22.54     .0780      .698,     1.000,      .0227, !
 23.80     .0857      .736,     1.000,      .0386, !
 24.09     .0480      .745,      .184,      .0112, TRIDECANE:
 24.45     .0697      .756,     1.000,      .0601, !
 24.75     .1095      .765,     1.000,      .0678, !
 25.36     .1001      .785,     1.000,      .0011, 2-CL-4-6-6-N-TRIAZINE:
 25.84     .0855      .800,     1.000,      .0556, !
 26.20     .2183      .810,      .184,      .0760, TETRADECANE:
 26.78    1.0287      .828,      .412,      .2602, DIMETHYLPHTHALATE:
 27.20    1.1559      .839,      .210,      .0182, DI-T-BU-B-NITROBENZENE:
 27.74     .3777      .843,     1.000,      .1726, !
 27.87     .2406      .865,      .184,      .0275, TETRADECANE:
 28.50     .2017      .884,     1.000,      .0922, !
 28.87     .1012      .895,     1.000,      .0463, !
 29.11     .1805      .900,      .314,      .0220, DIETHYLPHTHALATE:
 29.18     .0740      .903,      .414,      .0060, CL2 NORFLURAZON:
 29.65     .2876      .916,     1.000,      .0871, DS-ETHYL ATRATILE:
 30.65     .2058      .951,      .414,      .0551, CLPHOSPHENE ISOMER:
 30.85    1.7249     1.000,     1.000,     ATRAZINE:
 31.69     .3758      .979,      .314,      .0761, DIBUTYL PHTHALATE:
 32.07     .2585     1.003,     1.000,      .0459, !
 32.60     .0548     1.343,     1.000,      .0412, !
 36.27     .0258     1.061,     1.000,      .0150, !
 36.59     .1447     1.071,     1.000,      .1034, !
 36.99     .0858     1.000,      .735,      .1964, LASSO -- ALACHLOR:
 37.14     .4150     1.103,      .184,      .0810, DIBUTYL PHTHALATE:
 37.51     .5095     1.113,      .818,      .3593, ALASSO:
 38.38     .4254     1.139,      .215,      .2780, LASSO CL HOMOLOG:
 38.84     .0841     1.152,     1.000,      .0336, !
 39.08     .1093     1.159,     1.000,      .0680, !
 39.21     .2694     1.162,     1.000,      .0400, !
 39.70     .1261     1.206,      .735,      .0549, ALACHLOR:
 39.91     .1105     1.212,      .411,      .0429, DIELDRIN:
 39.85     .1092     1.235,      .258,      .0302, DIHEXYL PHTHALATE:
 40.27     .1840     1.248,     1.000,      .0897, !
 40.41     .1976     1.251,     1.000,      .1223, !
 41.02     .0937     1.272,     1.000,      .0583, !
 41.37    3.1135     1.283,      .325,      .6360, BENZYL BUTYL PHTHALA
 42.43     .2568     1.317,      .045,      .1198, TRIPHENYL PHOSPHATE:
 44.22     .4548     1.371,      .359,      .0794, DI-2-ET HEXYL PHTHALA
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Figure 1. GC computer print-out for Carrollton 70-year CCF.
RRT = relative retention time, RF = response factor (FID),
and C = concentration in µg/l.

the case of some herbicides, from the EPA Registration Division, Chemistry Branch, Pesticides Reference Standard Section in Washington. 1,2,3,4,5,7,7-Heptachloronorbornene was obtained from John Roach, Industrial Chemicals Contaminants Branch, Bureau of Foods, FDA, Washington. Deethylatrazine was obtained from the Ciba-Geigy Corp. 1,3,5-Trimethyl-2,4,6-trioxohexahydrotriazine (trimethyl isocyanurate) was synthesized using a two-step procedure starting with cyanuric chloride[5] by R. G. Webb of the ACB. The melting point and the IR and mass spectra of the product matched reported values.

RECOVERY STUDIES

The recovery of atrazine from water was studied to determine the approximate recoveries that might be expected for the atrazine-type herbicides. Four extraction methods were tested with spiked samples:

(a) Liquid-liquid extraction--Two 1-liter tap water samples were spiked with 100 and 200 µg, respectively, of atrazine, allowed to stand 5 days in laboratory light, and extracted 3 times with 100 ml of methylene chloride. The extracts were evaporated to 1 ml using a Kuderna-Danish evaporator followed by a slow stream of dry nitrogen, and analyzed by GC.

(b) Activated cocoanut charcoal (an incidental laboratory supply)--Five liters of tap water was spiked with 100 µg of atrazine and passed by gravity flow through a column of 11 grams (20-ml volume) of the charcoal (6-14 mesh). A control consisted of a similar charcoal column and 5 liters of unspiked water. For a direct recovery test, 100 µg of atrazine in 2 ml of chloroform was added directly to 11 grams of the same charcoal. The 3 charcoal samples were air-dried at room temperature overnight, then extracted with methylene chloride in a Soxhlet extractor for 6 hours. The extracts were evaporated to 1 ml as in experiment (a), and analyzed by GC.

(c) "Fine" mesh carbon (used at New Orleans)--Experiment (b) was repeated with 3.5 grams (20-ml volume) of the "fine" mesh Nuchar 190 carbon used in the New Orleans study.

(d) "Coarse" mesh carbon (used at New Orleans)--Experiment (b) was repeated with 10 grams (20-ml volume) of the "coarse" mesh carbon used for the New Orleans study.

Duplicates were run beginning with the atrazine spiked water.

RESULTS

The results of this New Orleans area drinking water supply analysis are summarized in Table 2 (at the end of the report). A total of 82 organic compounds were tentatively identified by GC-MS in one or more of the three finished drinking waters. Fifty-five of these identifications were confirmed. Nine compounds were not identified as to specific isomer, and eight others were identified only partially by certain structural features. Concentrations ranged from about 0.004 µg/l (the detection limit for most compounds except endrin) to 12 µg/l. Table 3 is a classification of the 82 compounds by chemical family or by use.

Table 3. Classification of Organics in New Orleans Drinking Water

Chemical Family or Use	Compounds Present	
Halogenated aliphatics	18	
Chlorinated aromatics	5	
Chlorinated aliphatic ethers	2	[a]
Herbicides & associated compounds	8	[b]
Pesticides	5	[c]
Aromatic hydrocarbons	11	
Aliphatic hydrocarbons	9	
Phthalates	11	
Miscellaneous	13	
Total	82	

[a] bis-2-chloroethyl ether and bis-2-chloroisopropyl ether

[b] Alachlor, alachlor homolog, atrazine, deethylatrazine, butachlor, cyanazine, propazine, and simazine.

[c] α-Chlordane, chlordene, DDE, dieldrin, and endrin

The 8 compounds only partially identified are listed as unknowns at the bottom of Table 2 and consist of 2 dichlorinated compounds (probably aromatics), 3 phthalates,

1 compound of apparent molecular weight 145, and 2 chlorinated-fluorinated hydrocarbons. In addition, 1 each C_2, C_3, and C_4 alkylbenzene compounds, 2 branched hydrocarbons (decane and undecane), a trimethylbenzene, an alachlor homolog, a dibromodichloroethane, and a heptachloronorbornene compound were not identified as to specific isomer.

No polynuclear aromatic hydrocarbons were identified in the carbon chloroform extracts, even after fractionation by TLC (detection limit is estimated at 0.1 µg/l after TLC separation), but naphthalene and 1- or 2-methylnaphthalene were identified in one XAD-resin extract.

No carboxylic acids or phenols were identified in any of the extracts, including the carbon alcohol extract, which was examined before and after methylation by diazomethane.

An independent investigator examined New Orleans drinking water and the same CCE extracts used for this study for volatile N-nitrosamines, but none were detected at 1 ng/l or above.[6]

α-Chlordane, chlordene, and pentachlorophenyl methyl ether were identified only qualitatively in the Carrollton mega-sample by GC-MS of fractions separated by column chromotography (see Experimental section). Endrin was detected and quantitated by electron capture GC of the appropriate fraction. None of these 4 compounds was detected by GC-MS analysis of the total extracts, in contrast to most of the compounds in Table 2.

Butyl octyl maleate, m-chloronitrobenzene, DDE, dicyclopentadiene, methylnaphthalene, and naphthalene were detected and confirmed by Junk at Ames, Iowa, during his independent GC-MS analysis of the Carrollton and Jefferson No. 1 resin extracts. They were not quantitated, and were not detected in the CCE's analyzed at SERL.

Using the VOA technique[7], which has proven excellent for very volatile organics in water, the 12 compounds listed below[8], not contained in Table 2, were identified only at the EPA's Water Supply Research Laboratory (WSRL), Cincinnati.

acetaldehyde	dichloromethane
acetone	ethanol
2-butanone	methanol
carbon disulfide	3-methyl butanal
carbon tetrachloride	2-methyl propanal

1,2-dichloroethane (8 µg/l) 1,1,2-trichloroethylene

Chloroform, identified at SERL by the tetralin extraction method, was detected at 133 µg/l by the WSRL. Five other compounds identified at SERL were also identified by the VOA technique at the WSRL (see footnotes to Table 2).

DISCUSSION

SEPARATION EXPERIMENTS

Organic analyses at SERL were performed using concentrated extracts from carbon and XAD-2 resin adsorption columns. Various attempts were made to fractionate the extracts to improve subsequent GC peak separation--most of these were unsuccessful (see Experimental Section).

The EPA column chromatographic method for organochlorine pesticides[1] was useful for the detection and quantitation of endrin and dieldrin, and for the detection of chlordene, α-chlordane, and pentachlorophenyl methyl ether. With the exception of dieldrin, these compounds were present at concentrations too low for GC-MS detection using the total extracts. Dieldrin was detected in the total extracts only after searching for characteristic peaks by limited mass range mass spectral techniques.

Thin layer chromatography was successful in fractionating the mega-sample CCE. Eighteen of the compounds identified by GC-MS analysis of the total extracts were also identified by GC-MS analysis of the eluates from the three regions of the developed TLC plots. However, little was gained by the additional TLC fractionation.

Fortunately, extract fractionation was not necessary for qualitative or even quantitative analysis. Computer controlled GC-MS analysis, with computer manipulation of acquired data, allowed identification of compounds by direct injection of CCE's. Figure 7 includes a TIC chromatogram of the unfractionated Carrollton 70-year CCE. Chromatograms of the Jefferson Co. 1 and Jefferson Co. 2 CCE's were similar. At this stage of separation, extracts were considered to be ready for GC-MS analysis.

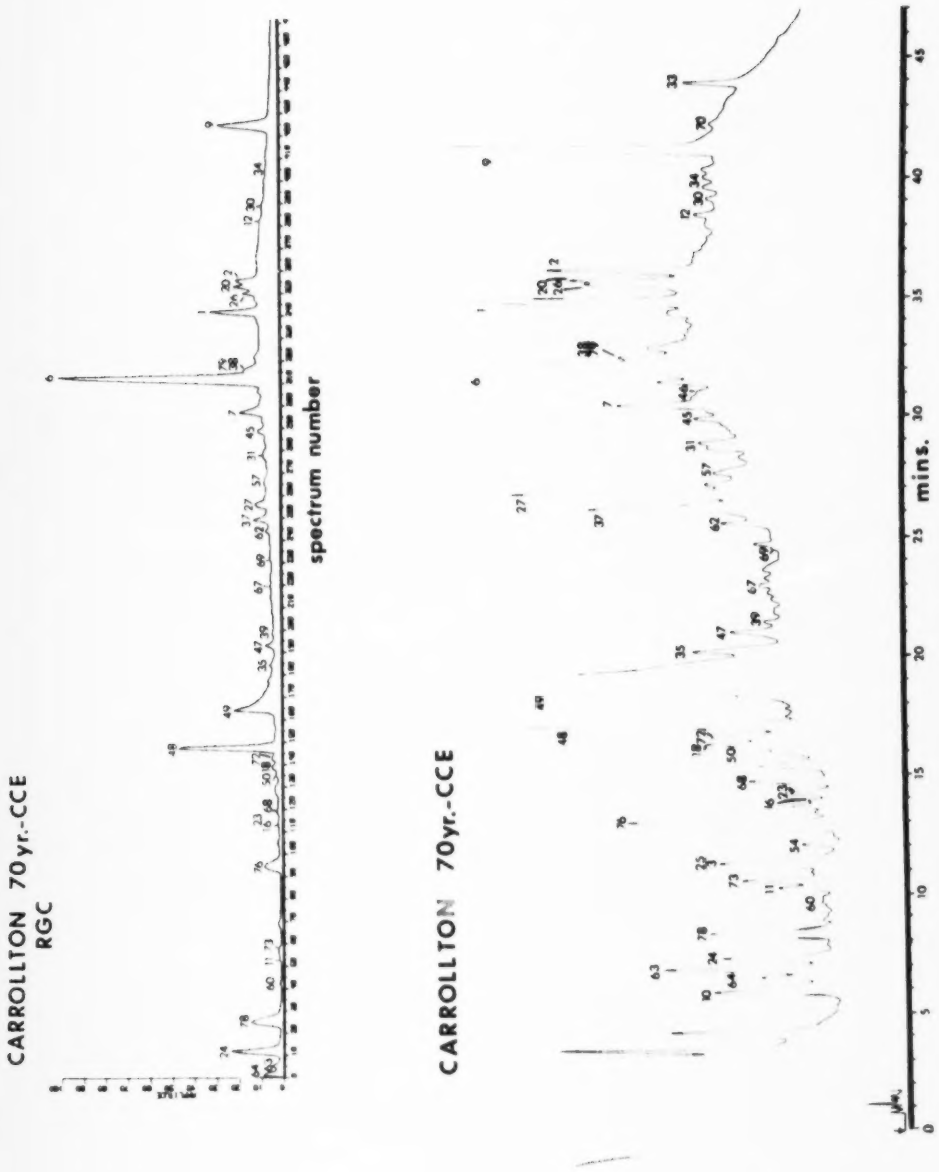

Figure 2. Reconstructed gas chromatogram (RGC) and FID gas chromatogram for Carrollton 70-year CCE. Numbers correspond to compounds in Table 2.

IDENTIFICATION OF COMPOUNDS

The initial output of the GC-MS-Computer system is a computer reconstructed gas chromatogram (RGC), which is a recording of the MS operating as a GC detector, and must ultimately be correlated with the FID chromatogram of the same sample. The RGC and FID chromatograms for the Carrollton 70-year CCE are shown in Figure 2. Peaks are numbered to correspond with the alphabetical listing of compounds in Table 2. High quality mass spectra, necessary to identify the peaks, were obtained using a separate PDP-8 computer with CRT and hard copier to obtain copies of the disk-stored mass spectra of sample components. In so doing, spectrum numbers corresponding to RGC peaks and a background subtraction routine were used to obtain the best possible spectra for all RGC peaks and shoulders.[5]

The first step in compound identification was an attempt to match each disk-stored sample spectrum with a spectrum in the EPA-Battelle (Columbus) computer file of 9500 mass spectra by direct acousticoupler-computer connection.[9] This was followed by a manual search of the Aldermaston Eight Peak Index of Mass Spectra,[10] regardless of the degree of computer match, or "Similarity Index". Compounds having a high Similarity Index (0.4 or above) with the computer selection and matching the peak listing in the eight-peak index or some other literature source, were considered "tentatively identified" with a high level of confidence. Compounds whose spectra matched the eight-peak index or other literature source, but whose spectra were not in the EPA-Battelle file, were also considered as "tentatively identified", but with a lower level of confidence.

Alachlor, cyanazine, and butachlor were identified by searching a herbicide handbook[11] for compounds of the same molecular weight and chemical family as indicated by examination of the mass spectra. These spectra were then matched with those of the pure compounds, samples of which were obtained from the EPA Pesticides Reference Standards Section.

Several compounds were identified both in a sample extract and in its blank. These were usually not considered as being in New Orleans water and are therefore not listed in Table 2. However, some compounds present in the blank, especially the mega-sample blank, at trace levels were present in the sample extract at more than five times the concentration in the blank. In such cases, the blank

concentrations were subtracted from the sample concentrations to give the quantitative values in Table 2.

Spectral Matching by Computer

About 75 apparently usable mass spectra were obtained from the Carrollton 70-year CCE. From these spectra 44 compounds were subsequently identified (not counting partially identified "unknowns"). The spectra of 17 compounds in the EPA-Battelle file were listed as "first hits" (best matches) in spectral searches with the usable sample spectra and later were determined to be correct identifications by comparison with standards. The similarity indices of 14 of the "first hits" were in the 0.4-0.8 range. Two were about 0.3, and one, dieldrin, was 0.216. Dieldrin, present at only 0.04 µg/l, gave a spectrum with many extraneous peaks, resulting in the low similarity index.

After spectra were checked against the EPA-Battelle file and Eight Peak Index of Mass Spectra[10], remaining unknown spectra were compared with those in the NIH Mass Spectral Search System computer file in Washington. Three or four compounds were identified by this comparison.

The EPA and NIH search systems have recently been combined into the Mass Spectral Search System file, handled by the Cyphernetics Corp., Ann Arbor, Michigan, and updated to include a total of 35,000 reference mass spectra.[9] This combination and expansion has resulted in a much more useful spectral matching system.

Limited Mass Range Searches

Simazine and propazine have almost the same GC retention time as atrazine, and since atrazine was present at much greater concentrations in most samples, its GC peak obscured the smaller simazine and propazine peaks. Indications of their presence were obtained by observations of their molecular ion peaks and molecular ion-minus-methyl peaks in the atrazine mass spectrum. Limited mass range mass spectrometry, in which the disk-stored mass spectral data are searched for ions that are definitive for a certain compound, produced evidence that simazine was present in the leading edge of the atrazine peak, and propazine in the trailing edge.

The limited mass range technique was also useful in searching the stored data for the spectrum of dieldrin, which did not produce a discernible peak on the computer

reconstructed gas chromatogram. Other compounds of special interest--bis-chloromethyl ether, geosmin, 2-methyl isoborneol, and deisopropylatrazine--were searched for by this technique in the Carrollton 70-year CCE, but were not detected.

Identification and Confirmation of Alachlor by Advanced Instrumentation

Alachlor, MW 269, does not give an easily discernible molecular ion peak by electron impact mass spectrometry, and its spectrum was not in the computer data bank, which made its identification difficult. The inability to identify the compound was particularly frustrating since its GC peak indicated that it was present in a relatively high concentration. Also, an associated peak in the gas chromatogram had a similar mass spectrum. Chemical ionization mass spectrometry, however, gave a characteristic molecular-ion-plus-one peak (m/e 270) for the compound (Figure 3). High resolution mass spectrometry, using the GC effluent, produced possible empirical formulae of the important mass fragments, including the molecular ion. These empirical formulae were correlated with reasonable fragmentation modes of the parent molecule, so that the most probable parent ion formula was deduced (Figure 3). Since other herbicides had been identified in the samples, the herbicide handbook[11] was searched for compounds having the appropriate molecular formula and structural characteristics. Alachlor was the only possibility. A standard of alachlor produced the same low resolution mass spectrum and GC retention time, confirming the identification.

The identity of alachlor was also confirmed by Fourier transform gas chromatography-infrared spectroscopy (see Figure 3), with a spectrum obtained on the fly from the Carrollton mega-sample extract injected into the GC. This spectrum matched that of the standard obtained in the same mode.

The electron impact spectrum of the alachlor homolog (compound 2, Table 2) did not give a definitive molecular ion. Chemical ionization MS showed the molecular weight to be 303 and the chlorine isotope pattern indicated the presence of two chlorine atoms instead of one. High resolution mass spectrometry supported this evidence, yielding an empirical formula of $C_{14}H_{19}NO_2Cl_2$. Mass spectrometry indicated the molecular structure to be the same as that of alachlor except for the presence of two chlorine atoms instead of one on the carbon atom alpha to

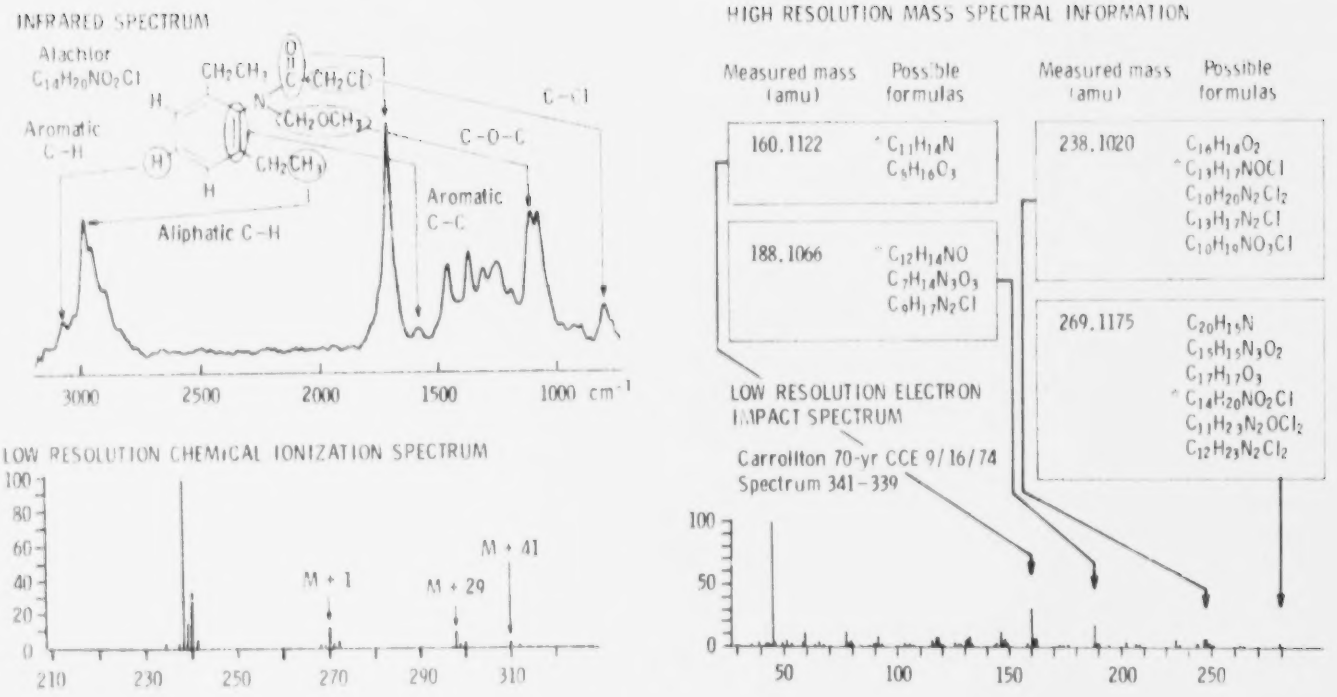

Figure 3. Spectra of alachlor in Carrollton 70-year CCE. Starred (*) formulas correspond to correct alachlor ion formulas.

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the carbonyl group. The Fourier transform GC-IR spectrum was very much like that of alachlor. On the basis of the above structural information the compound was tentatively identified as .2-dichloro-2',6'-diethyl-N-(methoxymethyl) acetanilide.

CONFIRMATION OF COMPOUNDS

After making tentative identifications (see under Identification of Compounds) attempts were made to confirm the identity of compounds in the drinking water by obtaining standards, matching GC retention times and mass spectra, and synthesizing compounds. Fifty-five compounds were confirmed in this way (Table 7). Mass spectra of selected compounds are shown in Figures 4 through 11. The degree of reliability of the "unconfirmed" identifications varies. Some compounds were identified by more than one method or in more than one laboratory. Also, most compounds were found in more than one of the drinking water supplies, and at the same sample site by more than one adsorption or extraction technique. Repeat findings indicate that such compounds are not analytical artifacts.

Alachlor, One Additional Chlorine Homolog

To confirm the identification of the alachlor homolog and to determine whether it could have been produced during chlorination treatment of New Orleans drinking water, solutions of 5 mg/l alachlor in distilled water were treated with up to 100 mg/l of chlorine gas for 22 hours at room temperature. Two chlorinated compounds were found, the concentrations of which were dependent upon the concentration of chlorine. The concentration of alachlor diminished correspondingly. These components were extracted with methylene chloride, which was then concentrated and analyzed by GC-MS. The spectra of these compounds did not match that of the alachlor homolog found in the New Orleans waters; they appeared to correspond to 1- and 7-chlorine additions to the aromatic ring of alachlor.

Therefore the homolog is probably not formed by chlorination of alachlor in the New Orleans water supply, but is possibly a by-product of alachlor manufacture.

Desethylatrazine

Desethylatrazine (desethylatrazine) was found in New Orleans water only in samples collected on carbon adsorption, not in resin adsorption samples. Wolfe and

Figure 4. Mass spectra of alachlor (top) and alachlor homolog, both from the Carrollton resin extract.

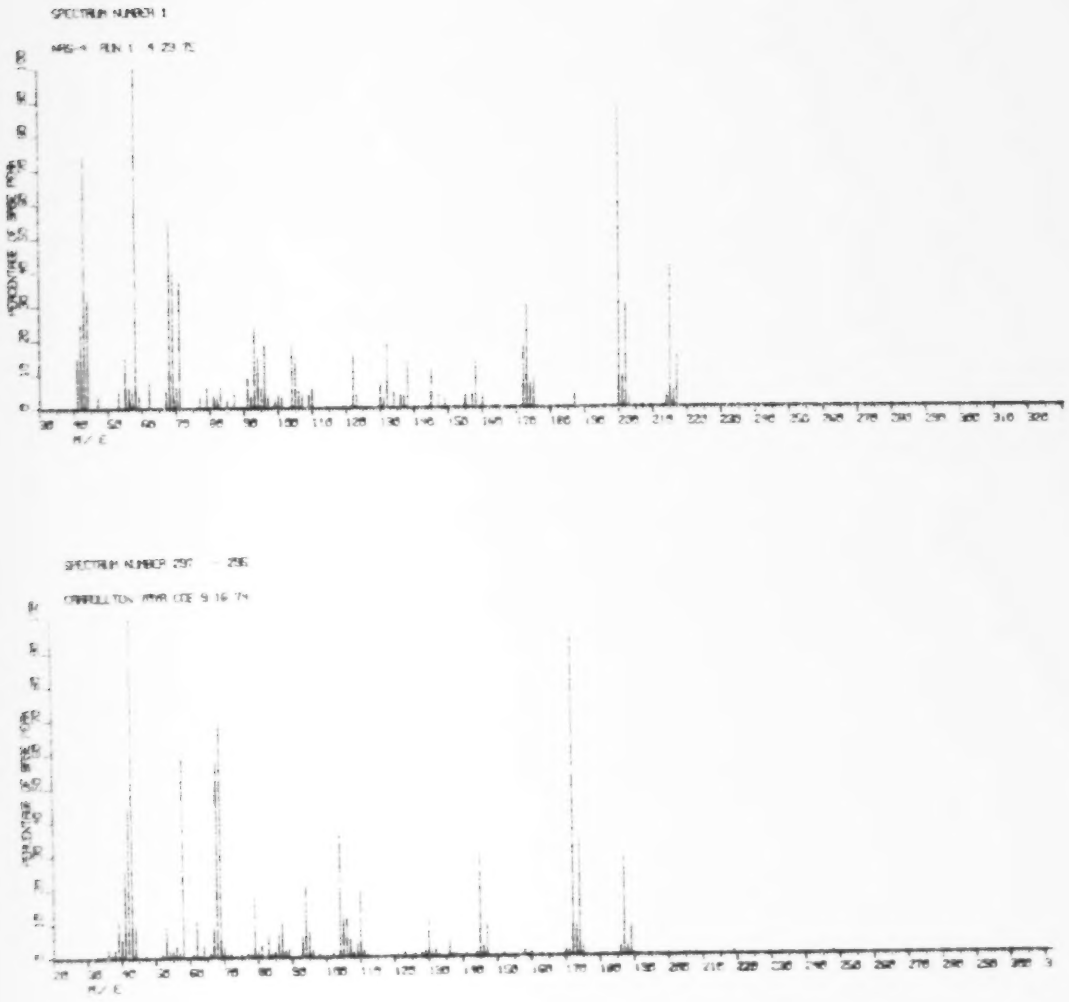


Figure 5. Mass spectra of atrazine standard (top) and deethylatrazine from Carrollton 70-year CCE.

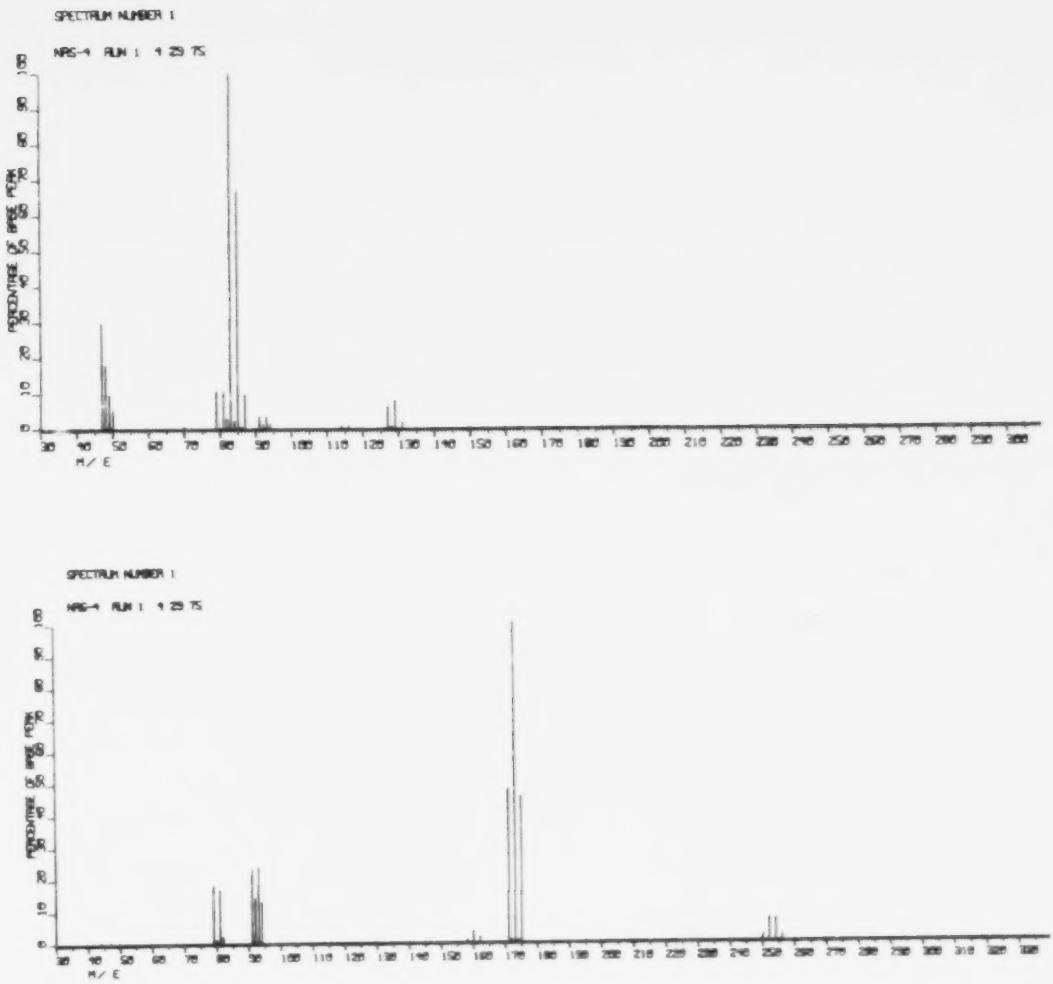


Figure 6. Mass spectra of bromodichloromethane (top) and bromoform, both standards.

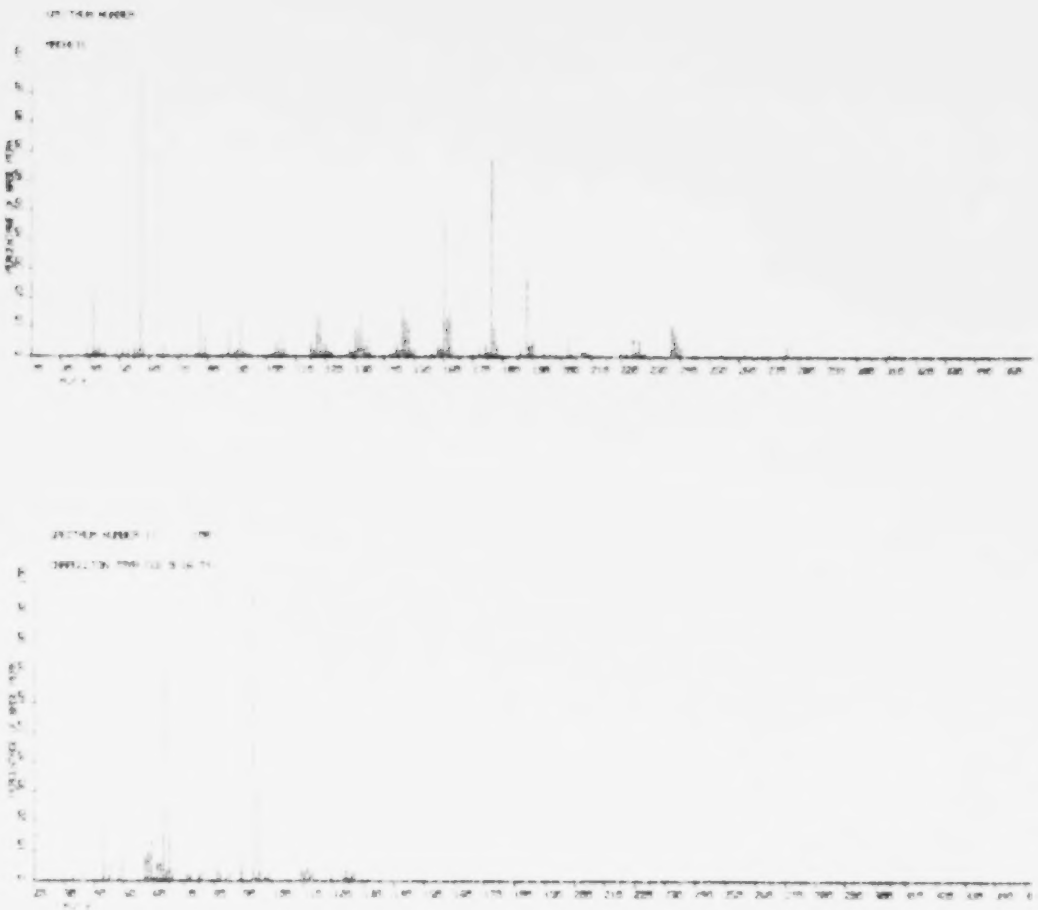


Figure 7. Mass spectra of butachlor (Machete®) standard (top) and bis-2-chloroethyl ether from the Carrollton 70-year CCB.

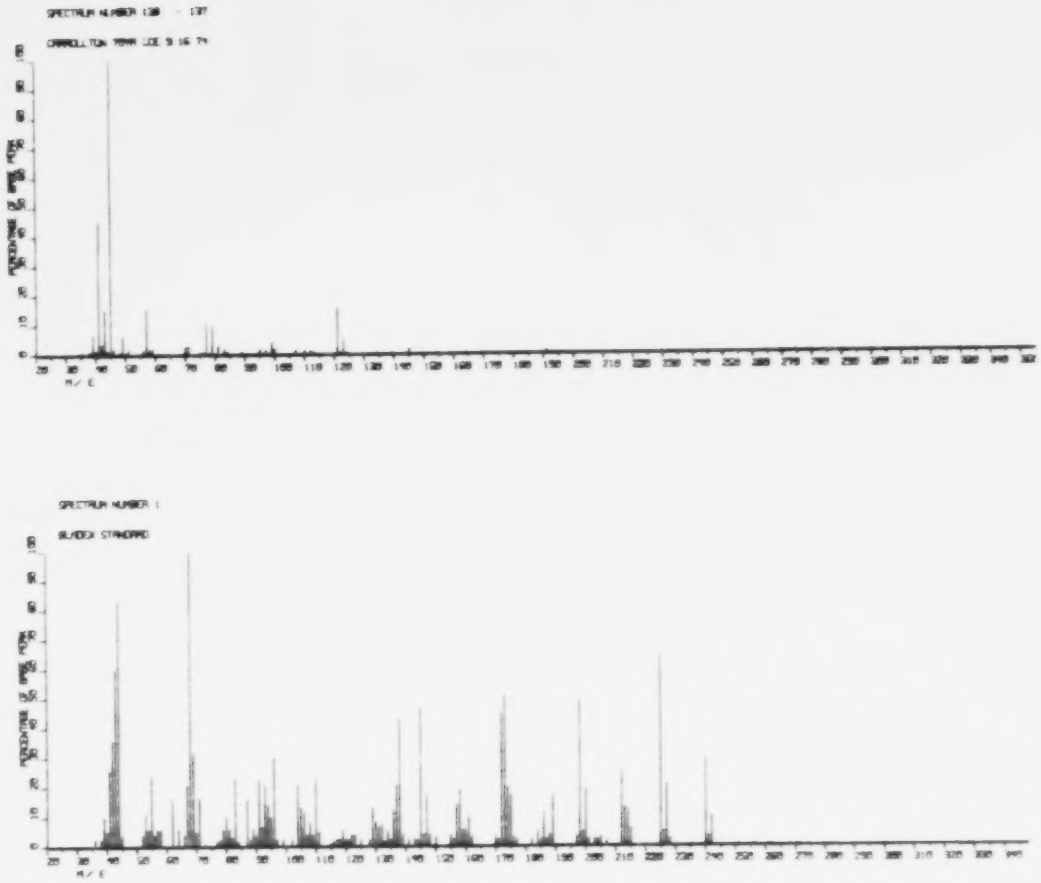


Figure 8. Mass spectra of bis-2-chloroisopropyl ether from the Carrollton 70-year CCE (top) and cyanazine (Bladex®) standard.

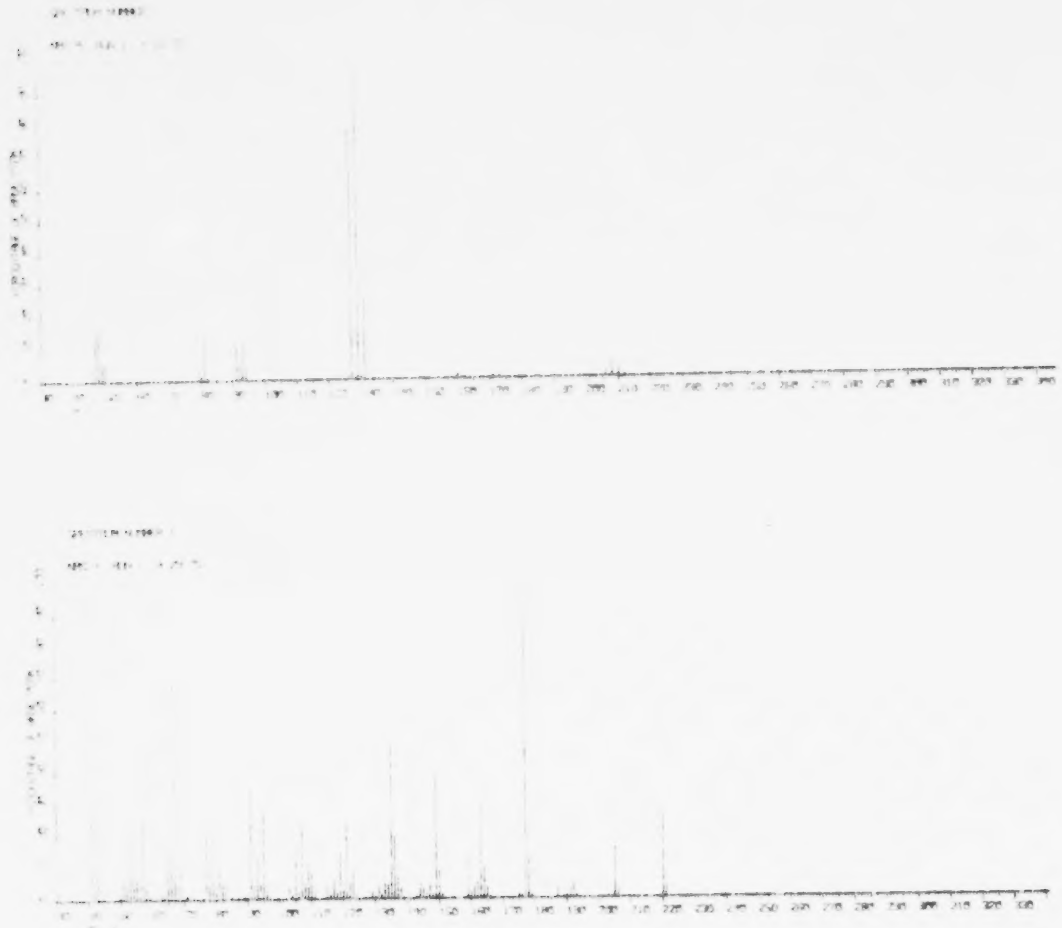


Figure 9. Mass spectra of dibromochloromethane (top) and 2,6-di-t-butyl-p-benzoquinone, both standards.

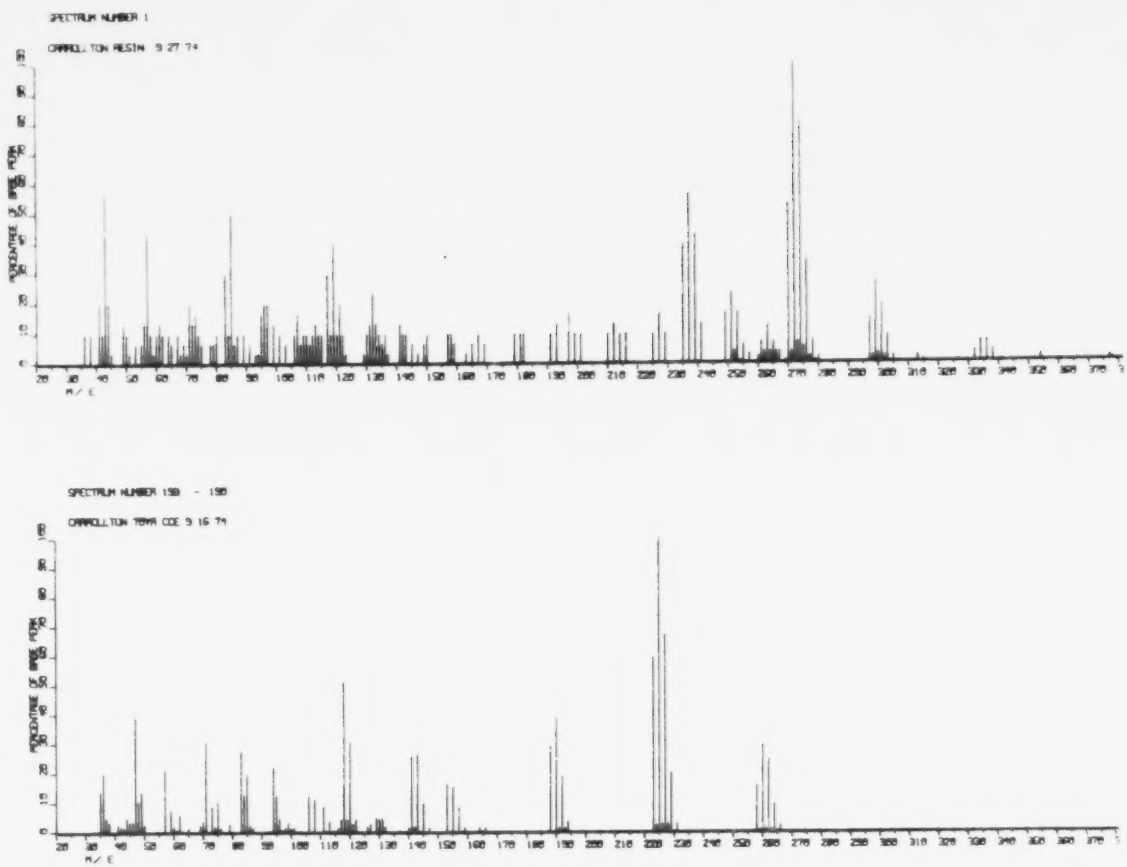


Figure 10. Mass spectra of 1,2,3,4,5,7,7-heptachloro-norbornene from the Carrollton resin extract (top) and hexachloro-1,3-butadiene from the Carrollton 70-year CCE.

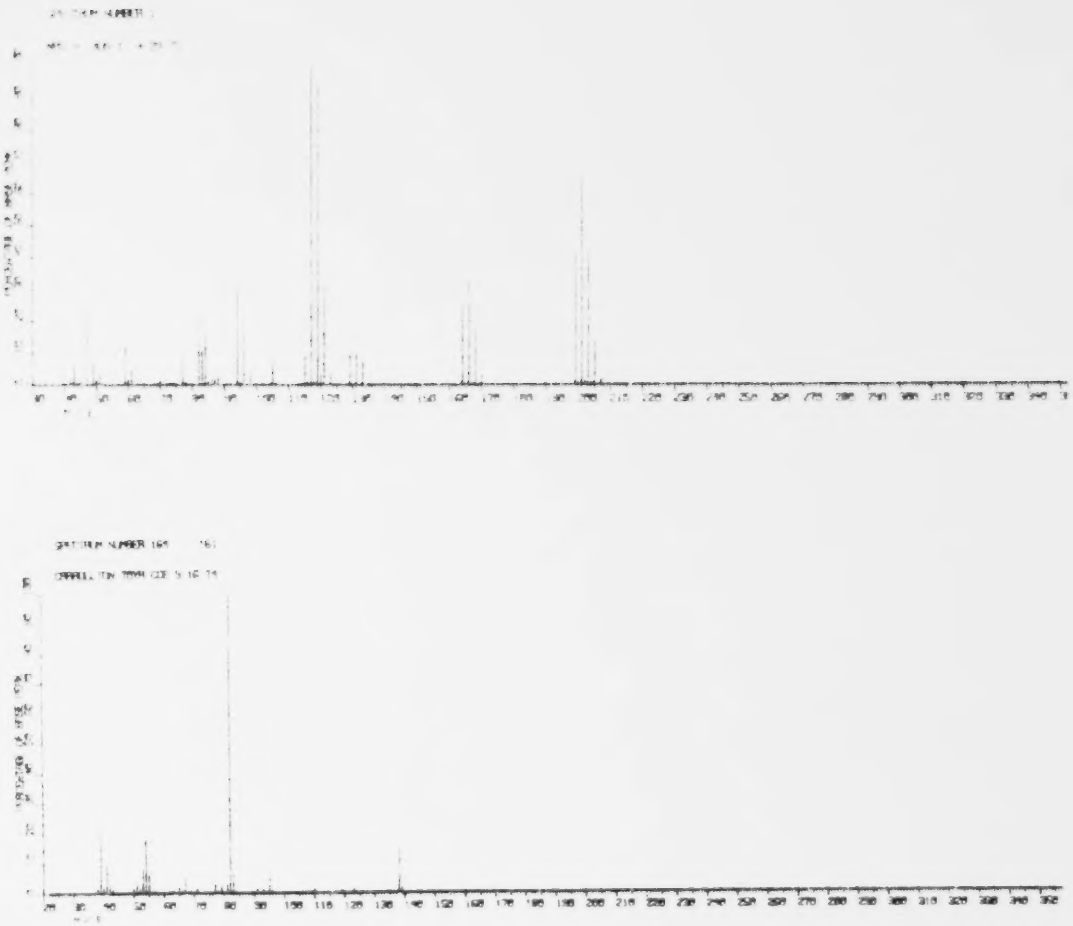


Figure 11. Mass spectra of hexachloroethane standard (top) and isophorone from the Carrollton 70-year CCB.

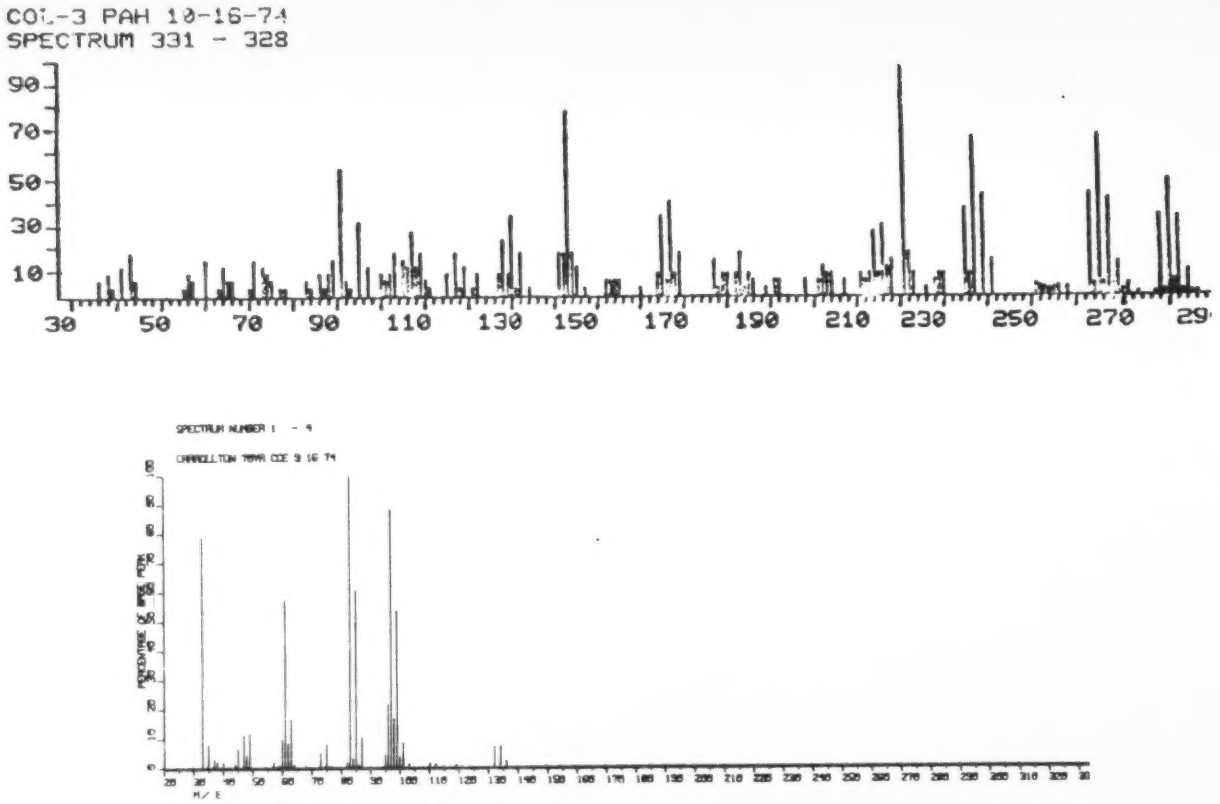


Figure 12. Mass spectra of pentachlorophenyl methyl ether from the Carrollton Mega Sample CCE (top) and 1,1,2-trichloroethane from the Carrollton 70-year CCE.

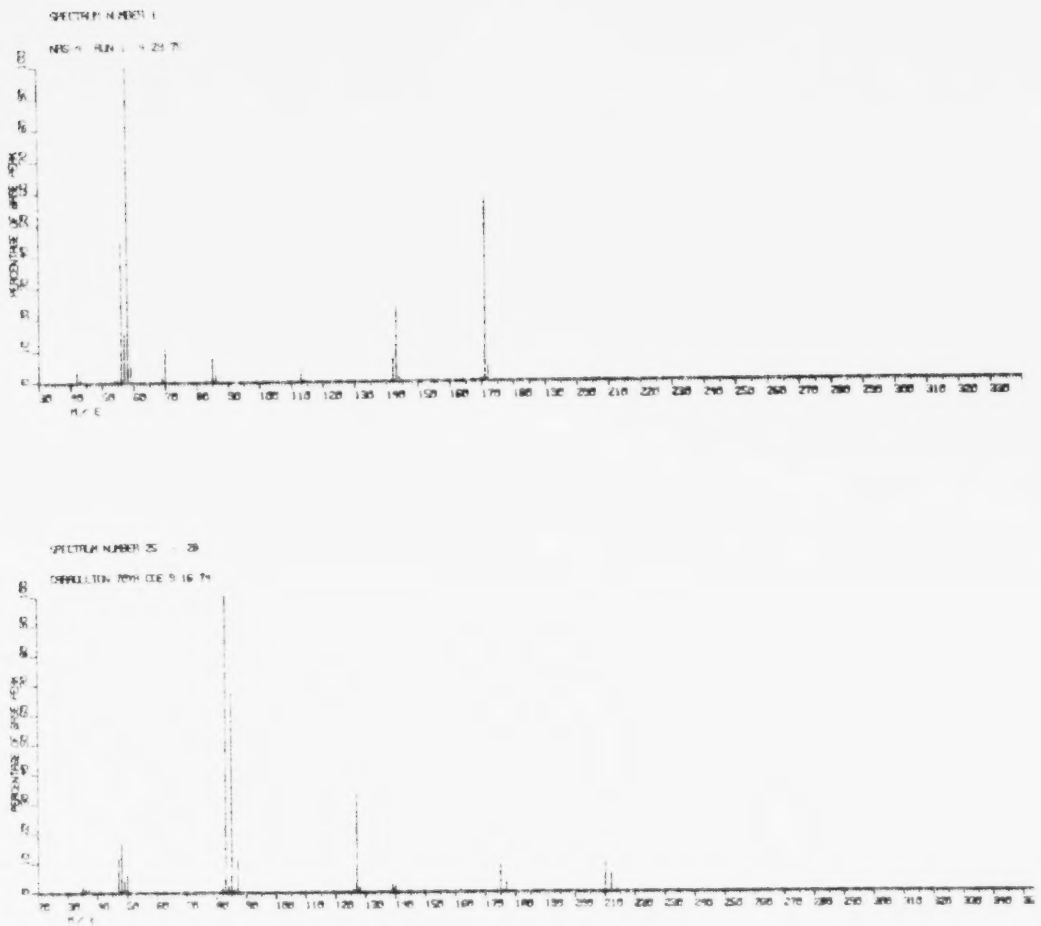


Figure 13. Mass spectra of 1,3,5-trimethyl-2.4,6-trioxo-hexahydrotriazine standard (top) and dichloroiodomethane from the Carrollton 70-year CCE.

Zepp[12] reported that deethylatrazine was the major photochemical degradation product of N-nitrosoatrazine in aqueous and chloroform solutions, but not in ethyl ether (which is used for eluting organics from the XAD-resin). Therefore, experiments were conducted to determine whether deethylatrazine could be produced by degradation of atrazine or of N-nitrosoatrazine on carbon or on the resin when exposed to varying intensities of light. Only atrazine was recovered from any of the experiments. Unfortunately, N-nitrosoatrazine decomposes to atrazine in a GC, the separation method used in these analyses. Repeated MS data, however, indicate that deethylatrazine is present in the New Orleans finished water, and is not an analytical artifact. It simply may not be adsorbed by XAD-resin, or may not elute from the resin.

1,3,5-Trimethyl-2,4,6-trioxohexahydrotriazine

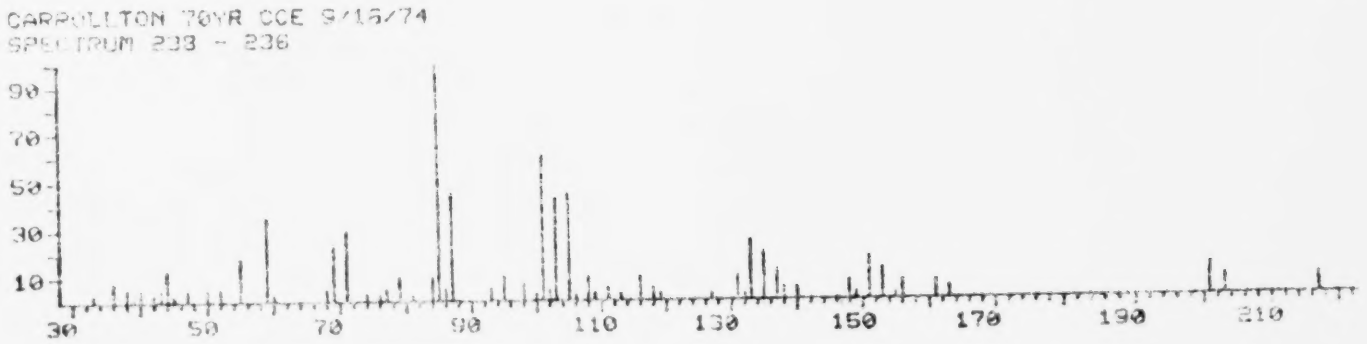
1,3,5-Trimethyl-2,4,6-trioxohexahydrotriazine (trimethyl isocyanurate), was tentatively identified by the Mass Spectral Search System, using the EPA-Biemann search mode (Similarity Index = 0.509). It is not available commercially and had to be prepared for confirmation (Experimental section). The mass spectra and GC retention time of the product matched that of the sample component, confirming the identification.

UNIDENTIFIED COMPOUNDS

Fifteen compounds at low concentrations (between 0.1 and 0.3 µg/l) yielded usable mass spectra, but could not be identified. Many other compounds in even lower concentrations were detected by GC, but did not give usable mass spectra. About 100 peaks can be counted in the gas chromatogram of the Carrollton 70-year CCE #1, but only 47 were identified.

Mass spectra of selected unidentified compounds in the New Orleans drinking water are shown in Figures 14 and 15.

In the New Orleans water samples, as in many other samples we have analyzed, the number of detected compounds increases almost exponentially as the detection limit decreases. This trend is summarized in Table 4.



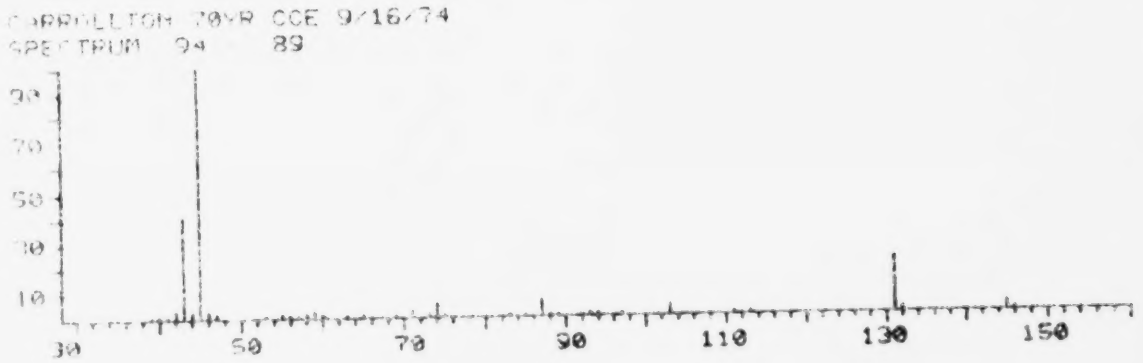


Figure 14. Mass spectra of unknown chlorinated fluorinated hydrocarbon (top) and unknown compound, apparent MW 145, both from the Carrollton 70-year CCE.

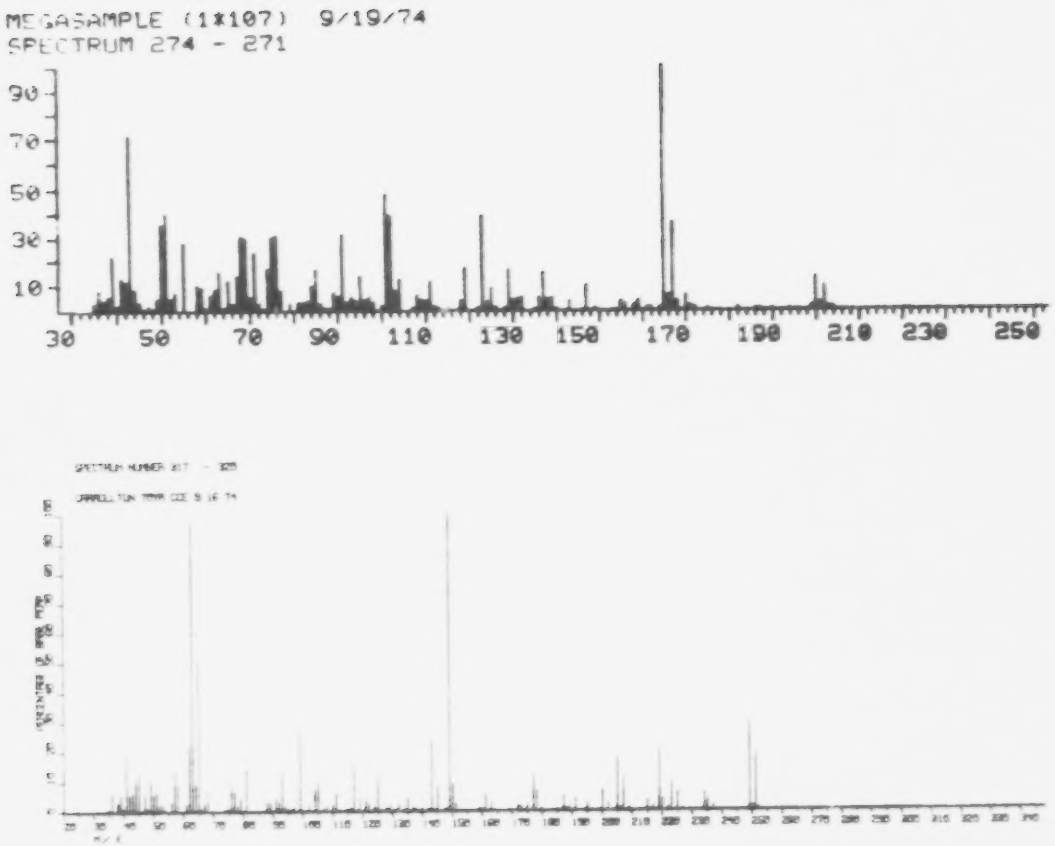


Figure 15. Mass spectra of unknown dichlorinated compound, MW 200, from the Carrollton Mega Sample CCE (top) and unknown dichlorinated compound, MW 249, from the Carrollton 70-year CCE.

Table 1. Classification of Organics in Carrollton CCE's by Concentration

Concentration Range, µg/l	Number of Compounds Present Within Concentration Range	
	70-year CCE	Mega-sample CCE
0.01 - 0.09	74*	91*
0.10 - 0.99	19	10
1.0 - 10	4	2
Total detected	97	103

*Includes compounds detected by FID/GC (manual peak count) but not identified.

Following this study, one FID gas chromatogram of the Carrollton 70-year CCE was obtained with a SERL-prepared glass capillary column-50 ft x 0.02 in. coated with SP-2100 (the packed column used 3% SP-2100). A total of 192 components were detected, about twice as many as were detected using the packed column, as a result of the lower detection limit caused by improved separation.

RECOVERIES

Several methods of extraction were used in these analyses--chloroform extraction of carbon filters in series, chloroform extraction of different sizes of carbon filters, alcohol extraction of carbon filters, liquid-liquid extraction with tetralin, and ether extraction of XAD-resin filters. The carbon filters in series gave better recoveries for the largest number of compounds.

In general, the concentrations for each of the three sampling sites are in good agreement for samples collected by the same techniques. For example, atrazine values for the 70-year CCE's are 4.9, 5.2, and 5.4 µg/l, and butachlor values are 0.05, 0.06, and 0.03 µg/l.

Recoveries from Carbon Filters in Series

Quantitative results in Table 2 for the 70-year CCE compounds are sums of the amounts recovered from two carbon

filters in series--filter #1 and filter #2. Table 5 gives for each compound at a concentration ≥ 0.05 µg/l the percent of the total concentration that was recovered in each of the two filters for the Carrollton 70-year sample.

Of the 29 compounds in Table 5, 24 were collected on filter #1 to the extent of 96% or more, indicating quantitative adsorption on carbon (but not quantitative recovery). These 24 include a wide variety of compounds, indicating a generally high degree of adsorption. However, there are exceptions. Three trihalogenated methanes or ethanes were adsorbed to the extent of only 64 to 85% on filter #1, and more hexachloroethane was found on filter #2 than #1.

Recoveries From Carbon Filters of Different Sizes

The two-month equivalent (mini-sample) CCE's contained much higher concentrations--up to 10 fold--of lower boiling compounds than did the 70-year and mega-sample CCE's, but about the same concentrations of the less volatile compounds. This may be seen by comparing the FID chromatogram of the Carrollton mini-sample CCE with that of the mega-sample, both in Figure 16, and with that of the 70-year sample in Figure 2. Although the 70-year sample was collected about ten days before the mini-sample, during which time the compound quantities in the river may have changed, the concentrations should not have changed as a function of volatility. These results may be an indication of organic overloading on the 70-year carbon filter, although this explanation is contrary to most of the results shown in Table 5. Seventy grams of carbon were used to extract 60 liters of water in the mini-sample filters (0.9 l/g), while 340 grams were used for 25,500 liters in the 70-year filters (75 l/g). This is an 83-fold difference in the water/carbon ratio.

Concentrations in the mega-sample CCE's were low relative to the 70-year CCE's. Of 30 compounds quantitated in the Carrollton drinking water by both methods, the concentrations of 21 in the mega-sample were between 25 and 76% of that found in the 70-year sample. The water/carbon ratio for the mega-sample was 57 l/g.

Several operational factors were biased against the mega-sample quantitations relative to those for the 70-year sample. The mega-sample carbon was dried for 10 days before extraction, while the 70-year sample carbon was dried for only 2 days. The mega-sample CCE was concentrated three times more than the 70-year CCE, and concentration was not

Table 5. Percent of Compounds Recovered from Carrollton 70-year Filters #1 and #2 in Series*

Compound	Total Concentration µg/l	% From Filter #1	% From Filter #2
Alachlor	0.82	97	3
Alachlor homolog	0.28	100	0
Atrazine	4.9	98	2
Atrazine, deethyl	0.53	96	4
Benzyl butyl phthalate	0.64	100	0
Bromoform	0.57	100	0
Butachlor	0.05	100	0
Cyanazine	0.35	100	0
Chlorodibromomethane	1.1	64	36
bis-2-Chloroethyl ether	0.04	100	0
bis-2-Chloroisopropyl ether	0.18	100	0
Dibromodichloroethane isomer	0.33	100	0
Dibutyl phthalate	0.10	80	20
2,6-Di-t-butyl-p-benzoquinone	0.22	100	0
Dichloroiodomethane	1.1	85	15
Dieldrin	0.04	100	0
Di-(2-ethylhexyl) phthalate	0.10	100	0
Dimethyl phthalate	0.27	100	0
Dipropyl phthalate	0.07	100	0
1,2,3,4,5,7,7-Heptachloronorbornene	0.06	100	0
Heptachloronorbornene isomer	0.06	100	0
Hexachloro-1,3-butadiene	0.16	100	0
Hexachloroethane	4.3	36	64
Isophorone	1.6	98	2
1,1,2-Trichloroethane	0.35	69	31
Trimethylbenzene isomer	0.04	100	0
Triphenyl phosphate	0.12	100	0
Undecane, branched	0.06	100	0
o-Xylene	0.33	100	0

*Including only compounds present in total concentration of 0.05 µg/l or greater.

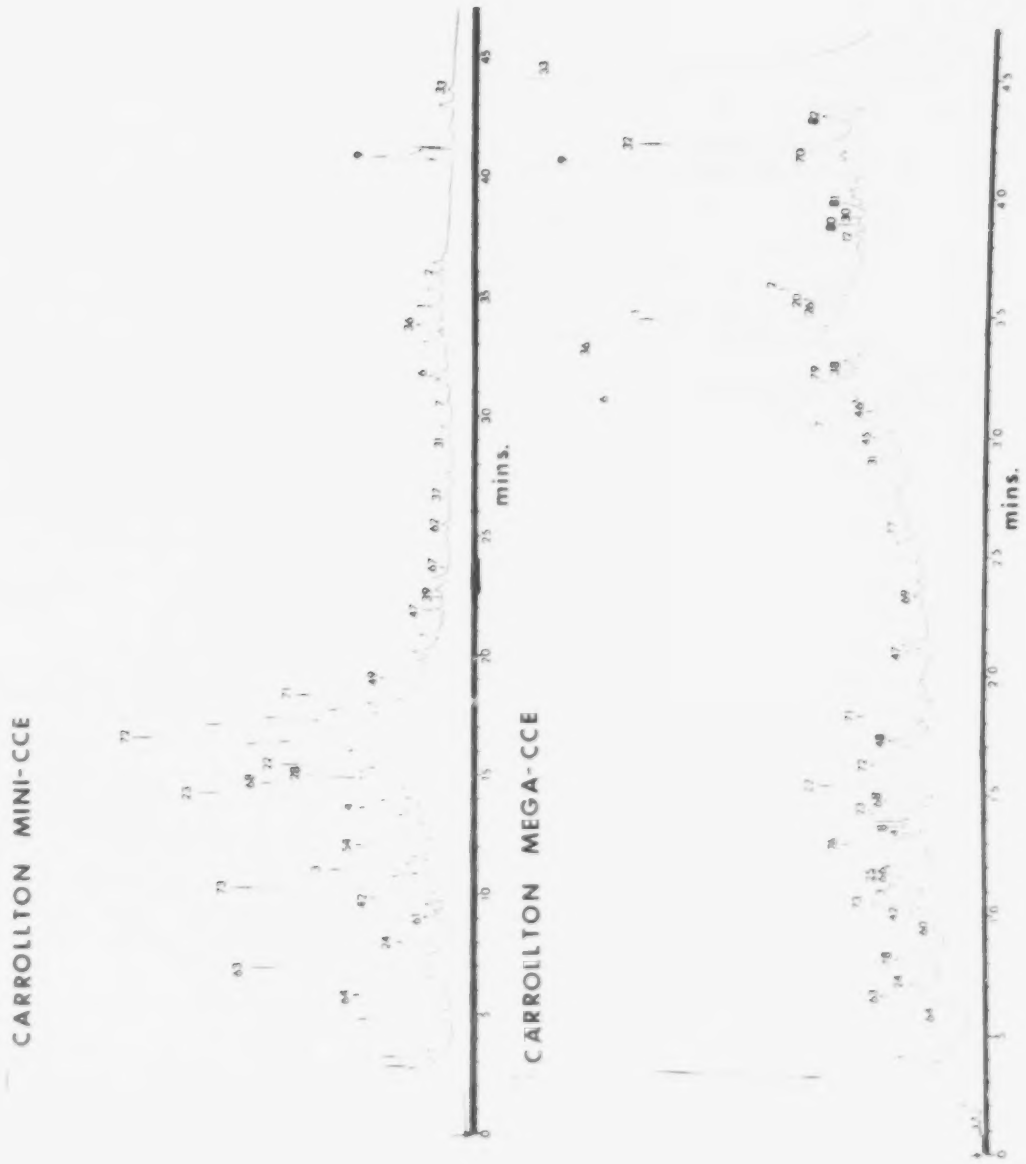


Figure 16. FID gas chromatograms for the Carrollton mini- and mega-CCE's. Numbers correspond to compounds in Table 2.

as carefully controlled, in that direct distillation of the chloroform was used for the mega-sample whereas vacuum evaporation at room temperature was used for the 70-year CCE's.

Carbon alcohol extract (CAE)

The methylated carbon-alcohol extract (Carrollton 70-year) was expected to contain carboxylic acids and phenols, but none were found. Only compounds 1, 6, 7, 37, 49, and 69 (Table 2), which were identified in the 70-year CCE, were found in the CAE. Of these, the one present in highest concentration was atrazine at 0.3 µg/l. (This is included in the value of 4.9 µg/l given for the CCE in Table 2.)

Recovery from resin

Not enough data are available from the New Orleans study to make a good comparison of XAD-resin vs. carbon extraction efficiencies. More compounds and higher concentrations were obtained from the carbon, but sampling circumstances were biased against the resin technique. A well-designed comparison of these two concentration techniques is needed.

Recoveries Using Tetralin Extraction

Chloroform was the only compound identified by the tetralin extraction technique. Only 2 or 3 GC peaks were observed that were not also in the tetralin blank. This technique is good for volatile compounds, since the high boiling solvent leaves a window for early eluting GC peaks, but its sensitivity is limited since tetralin cannot be evaporated to concentrate extracted pollutants. This especially limits the usefulness of tetralin in drinking water analysis. However, tetralin has now been purified by ZSDL to eliminate interference peaks so that 1 ml can be used to extract 2 liters of water, thus increasing the sensitivity. Although more complicated, the gas stripping VOA technique is far more productive for isolating these volatile compounds.

RECOVERY STUDIES WITH ATRAZINE

Results of the atrazine recovery experiment are summarized below:

Extraction with methylene chloride, 100 µg/l spike
Extraction with methylene chloride, 200 µg/l spike

Cocoanut charcoal +100 µg atrazine in 5 liters of water	29
Cocoanut charcoal +100 µg atrazine added directly	28
New Orleans fine carbon +100 µg atrazine in 5 liters of water	92
New Orleans fine carbon +100 µg atrazine added directly	103
New Orleans coarse carbon +100 µg atrazine in 5 liters of water	59
New Orleans coarse carbon +100 µg atrazine in 5 liters of water (Duplicate)	71
New Orleans coarse carbon +100 µg atrazine added directly	102

Liquid-liquid extraction with methylene chloride is a good method for recovery of atrazine from water, as is carbon adsorption with the fine grade carbon. In the New Orleans 70-year carbon samplers, the coarse carbon was used on each end of the column as a filter and holder for the fine grade carbon, which was assumed to be the main adsorbing material.

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TABLE 2

ORGANIC COMPOUNDS IN NEW ORLEANS DRINKING WATER--CONCENTRATIONS IN µg/l**

	COMPOUND	KRV[+]	CARROLLTON WATER PLANT				JEFFERSON #1 WATER PLANT			JEFFERSON #2 WATER PLANT			Notes
			70-Year[++] CCE	2-Month CCE	Mean-Sample CCE	Resin Extract	70-Year[++] CCE	2-Month CCE	Resin Extract	70-Year[++] CCE	2-Month CCE	Resin Extract	
1	Alachlor* (Lasso®) (2-chloro-2',6'-diethyl-N-(methoxymethyl)acetanilide)	1.09	0.67	1.9	0.44	0.67	2.3	2.9	0.35	2.1	1.4	0.17	c,i,f,g,j,i,j
2	Alachlor, one additional chlorine homolog	1.14	0.26	1.7	0.18	0.21		P		0.05	P		d,f,g,h,i
3	Alkylbenzene-C_8 (m-xylene or p-xylene)	0.35	0.03	7.5	0.05			5.6			6.2		
4	Alkylbenzene-C_9 (p-ethyltoluene or n-propylbenzene)	0.41		2.4	0.01			1.9		0.03	2.2		
5	Alkylbenzene-C_4	<0.02	<0.1			J							g
6	Atrazine* (2-chloro-4-ethylamino-6-isopropylamino-s-triazine)	1.00	4.0	3.7	2.7	1.0	5.2	4.8	0.64	3.4	3.2	0.18	c,i,d,f,g,h,i,j
7	Atrazine, deethyl* (2-chloro-4-amino-6-isopropylamino-s-triazine) (desethylatrazine)	0.96	0.31	0.78	0.22		0.27	0.80		0.27	0.75		c,g

Cont'd

TABLE 2 (Continued)

ORGANIC COMPOUNDS IN NEW ORLEANS DRINKING WATER--CONCENTRATION IN µg/l**

	COMPOUND	RRT+	CARROLLTON WATER PLANT				JEFFERSON #1 WATER PLANT			JEFFERSON #2 WATER PLANT			Notes
			70-Year++ CCE	2-Month CCE	Mega-Sample CCE	Resin Extract	70-Year++ CCE	2-Month CCE	Resin Extract	70-Year++ CCE	2-Month CCE	Resin Extract	
8	Benzaldehyde*	0.44			0.03								h
9	Benzyl butyl phthalate*	1.29	0.64	1.4	0.24		0.83	1.8		0.75	1.6	0.08	g,h
10	Bromodichloro-methane	0.18	P			<0.1			<0.1				b
11	Bromoform*	0.32	0.57										b,g
12	Butachlor* (Machete®) [2-chloro-2',6'-diethyl-N-(butoxymethyl)-acetanilide]	1.21	0.05		0.02	<0.1	0.06			0.05			g,h
13	Butyl octyl maleate*					J			J				
14	α-Chlordane				<0.1(T)								h
15	Chlordene*				<0.1(T)								h
16	bis-2-Chloroethyl ether*	0.44	0.04				0.16			0.12			g
17	Chloroform*												a,b
18	bis-2-Chloroisopropyl ether*	0.51	0.18		P		0.08			0.03			g,h
19	m-Chloronitrobenzene*					J			J				

TABLE 2 (Continued)

ORGANIC COMPOUNDS IN NEW ORLEANS DRINKING WATER--CONCENTRATION IN µg/l**

	COMPOUND	RRT+	CARROLLTON WATER PLANT				JEFFERSON #1 WATER PLANT			JEFFERSON #2 WATER PLANT			Notes
			70-Year++ CCE	2-Month CCE	Mega-Sample CCE	Resin Extract	70-Year++ CCE	2-Month CCE	Resin Extract	70-Year++ CCE	2-Month CCE	Resin Extract	
20	Cyanazine* (Bladex®) [2-(4-chloro-6-ethylamino-s-triazin-2-ylamino)-2-methylpropionitrile]	1.13	0.35		<0.01		0.21			0.31			g,h
21	DDE*[2,2-bis-(p-chlorophenyl)-1,1-dichloroethylene]					0.05(J)			0.05(J)				
22	n-Decane*	0.48		2.4	0.06			2.0			2.0		d
23	Decane, branched	0.44	0.02	5.8	0.03			5.4			5.2		
24	Dibromochloromethane*	0.23	1.1	<10	0.6	<0.1	0.4		<0.1	0.06	P		b,d,g
25	Dibromodichloroethane isomer	0.35	0.33		0.16					0.63			g
26	Dibutyl phthalate*	1.12	0.10		0.09	0.05	0.36		0.01	0.23		0.03	e,f,g
27	2,6-Di-t-butyl-p-benzoquinone*	0.84	0.22				0.21			0.25			
28	m-Dichlorobenzene*	0.46		<3					<0.1				f
29	Dicyclopentadiene*								J				
30	Dieldrin*	1.23	0.04		0.01	0.01(J)	0.07		0.01(J)	0.05			g,h
31	Diethyl phthalate*	0.91	0.03	0.24	0.02	J	0.03	0.10	0.03	0.01	0.18		e,f,g,h

TABLE 2 (Continued)

ORGANIC COMPOUNDS IN NEW ORLEANS DRINKING WATER--CONCENTRATION IN µg/l**

	COMPOUND	RRT+	CARROLLTON WATER PLANT				JEFFERSON #1 WATER PLANT			JEFFERSON #2 WATER PLANT			Notes
			70-Year++ CCE	2-Month CCE	Mega-Sample CCE	Resin Extract	70-Year++ CCE	2-Month CCE	Resin Extract	70-Year++ CCE	2-Month CCE	Resin Extract	
32	Di-(2-ethylhexyl) adipate*	1.32			0.10								
33	Di-(2-ethylhexyl) phthalate*	1.38	0.10	0.40	11	0.05	0.46	0.50	J	0.27	1.2	0.16	d,f,g
34	Dihexyl phthalate	1.24	0.03			0.05						0.16	g
35	Dihydrocarvone	0.63	0.14				0.06			0.07			
36	Diisobutyl phthalate*	1.06		<1	0.59	<0.05							f
37	Dimethyl phthalate*	0.83	0.27	0.60	P		0.13	0.82		0.18	0.74		c,f,g,h
38	Dipropyl phthalate*	1.02	0.07		0.01		0.13			0.14			g
39	n-Dodecane*	0.67	0.01	0.10				0.40			0.37		
40	Endrin*	1.27 (1st peak)			0.004 (T)		0.008 (T)			0.006 (T)			
41	Ethyl acetate					P						P	
42	Ethylbenzene	0.32		2.3	0.02			1.6			1.8		
43	o-Ethyltoluene*	0.45					0.04			0.02			
44	m-Ethyltoluene*	0.42					0.05			0.02			

TABLE 2 (Continued)

ORGANIC COMPOUNDS IN NEW ORLEANS DRINKING WATER--CONCENTRATIONS IN μg/l**

	Compound	RRT[b]	CARROLLTON WATER PLANT				JEFFERSON #1 WATER PLANT			JEFFERSON #2 WATER PLANT			Notes
			70-Year[a] CCE	2-Month CCE	Mega-Sample CCE	Resin Extract	70-Year[++] CCE	2-Month CCE	Resin Extract	70-Year[++] CCE	2-Month CCE	Resin Extract	
45	1,2,3,4,5,7,7-Heptachlorbornor-nene*	0.94	0.06		0.03	0.02	0.07		0.04	0.07		0.01	a,e, g,h
46	Hexachlorocyclobu-tene isomer	0.96	0.06		0.02		0.04			0.04			g,h
47	Hexachloro-1,3-butadiene*	0.65	0.16	0.70	0.07	0.04	0.27		0.12	0.21		<0.01	a,e, f,g,h
48	Hexachlorobenzene*	0.53	2.1		0.39	0.03	0.19		<0.1	0.30			f,g,h
49	Tetrachloroethene*	0.73	1.6	2.9			2.8	<11		2.9	9.5		e,g
50	Dimethene*	1.00	0.03										
51	Methyl benzoate*	0.60							<0.01				e
52	2-Methylnaphthalene*								J				
53	Naphthalene*					J			J				
54	n-Nonane*	0.38	0.03	2.4				2.4			2.1		
55	Pentachloroethane*	<0.38				<0.1							
56	Pentachlorophenyl methyl ether				<0.1 (E)								
57	n-Pentadecane*	0.86	0.03					0.10	0.01		0.10	<0.01	

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TABLE 2 (Continued)

ORGANIC COMPOUNDS IN NEW ORLEANS DRINKING WATER--CONCENTRATIONS IN µg/l**

	COMPOUND	RRT+	CARROLLTON WATER PLANT				JEFFERSON #1 WATER PLANT			JEFFERSON #2 WATER PLANT			Notes
			70-Year++ CCE	2-Month CCE	Mega-Sample CCE	Resin Extract	70-Year++ CCE	2-Month CCE	Resin Extract	70-Year++ CCE	2-Month CCE	Resin Extract	
58	Propazine* [2-chloro-4,6-bis-(isopropylamino)-s-triazine]	∿1.0			<0.1		<0.1			<0.1			
59	Simazine* [2-chloro-4,6-bis-(ethylamino)-s-triazine]	∿1.0	<0.1			J	<0.1			<0.1			g
60	1,1,1,2-Tetrachloroethane*	0.30	0.04		0.11								
61	Tetrachloroethylene*	0.26		<1		<0.1	0.20	<5	<0.1	0.20	<5		b,g
62	n-Tetradecane*	0.80	0.02	0.10				0.10			0.12		
63	Toluene*	0.22		11	0.08		0.10	7.1	<0.01		12		b
64	1,1,2-Trichloroethane*	0.21	0.35	6.2	<0.2	<0.1	0.45	8.5	<0.1	0.41	6.4		
65	1,1,1-Trichloropropane	∿0.07				<0.1							
66	1,2,3-Trichloropropane	0.36			<0.2								
								0.17			0.20		
67	n-Tridecane*	0.72	0.01	0.30									
68	Trimethylbenzene isomer	0.46	0.04	6.1	0.02	0.01		5.1		0.02	5.3		

TABLE 2 (Continued)

ORGANIC COMPOUNDS IN NEW ORLEANS DRINKING WATER--CONCENTRATIONS IN µg/l**

	COMPOUND	RRT+	CARROLLTON WATER PLANT				JEFFERSON #1 WATER PLANT			JEFFERSON #2 WATER PLANT			Notes
			70-Year++ CCE	2-Month CCE	Mega-Sample CCE	Resin Extract	70-Year++ CCE	2-Month CCE	Resin Extract	70-Year++ CCE	2-Month CCE.	Resin Extract	
69	1,3,5-Trimethyl-2,4,6-trioxo-hexahydrotriazine*	0.73	0.01		0.01								c,g
70	Triphenyl phosphate*	1.31	0.12		0.03								h
71	n-Undecane*	0.58		2.5	0.03			<10			2.1		
72	Undecane, branched	0.52	0.06	5.3	0.04								
73	o-Xylene*	0.33	0.33	4.1	0.12			2.8			3.4		
74	Unknown chlorinated fluorinated hydrocarbon	0.78	<0.01			<0.1			<0.1			<0.1	
75	Unknown chlorinated fluorinated hydrocarbon	0.82	<0.01			<0.1			<0.1			<0.1	
76	Unknown compound, apparent MW 145	0.40	<0.7		<0.3		<0.9			<0.9			g
77	Unknown dichlorinated compound MW 200	0.82			0.05								
78	Dichloroiodomethane	0.26	1.1		0.84		1.3			1.6			g,k
79	Unknown dichlorinated compound MW 249	1.0	<0.04		<0.02		P			P			

TABLE 2 (Continued)

ORGANIC COMPOUNDS IN NEW ORLEANS DRINKING WATER--CONCENTRATIONS IN µg/l**

	COMPOUND	RRT+	CARROLLTON WATER PLANT				JEFFERSON #1 WATER PLANT			JEFFERSON #2 WATER PLANT			Notes
			70-Year++ CCE	2-Month CCE	Mega-Sample CCE	Resin Extract	70-Year++ CCE	2-Month CCE	Resin Extract	70-Year++ CCE	2-Month CCE	Resin Extract	
80	Unknown phthalate	1.22			0.01								
81	Unknown phthalate	1.24			0.01								
82	Unknown phthalate	1.36			0.12								

* Confirmed by matching GC retention time and, in most cases, mass spectrum with that of a standard.

** Blank spaces indicate that compound was not detected in that specific sample.

\+ Relative retention time (to atrazine); mostly determined by the GC computer system.

++ Concentration values for 70-year CCE compounds are sums of concentrations in CCE #1 (filter #1) and CCE #2 (filter #2).

J Detected only by Junk, USAEC-Ames Laboratory. Identified in the Carrollton or Jefferson No. 1 resin extracts by GC-MS, but not quantitated except for DDE and dieldrin. These compounds were not detected at SERL in the resin extracts.

P Present, usually in very low concentrations (<0.05 µg/l), but not quantitated due to interferences with other GC peaks or lack of a discernable GC peak (detected only by mass spectrometry).

T Detected only by Thruston at SERL by GC-MS after fractionation by column chromatography.

NOTES:

a Chloroform was identified at SERL only in the tetralin extract of the Carrollton water.
b These compounds were also identified at the Water Supply Research Laboratory, EPA, Cincinnati, by VOA analysis of the Carrollton water.
c Also identified in the Carrollton CAE.
d Also identified by Junk, USAEC-Ames Laboratory, in the Carrollton resin extract.

Notes (Continued)

e Also identified by Junk, USAEC-Ames Laboratory, in the Jefferson #1 resin extract.

f Also identified by Melton at the Water Supply Research Laboratory, EPA, Cincinnati, by GC-MS analysis of a solvent extract of a 1 liter grab sample of the Carrollton water.

g Also identified by duplicate analysis of the Carrollton 70-year CCE using the Varian CH5/DF system.

h Also identified by GC-MS (Finnigan) analysis of the Carrollton mega-sample CCE after fractionation by TLC.

i Confirmed by high resolution mass spectrometry (only a structural indication in the case of compound #2).

j Confirmed by GC-Ft-IR.

k Identification based on a report received after this table was prepared (EPA Mass Spectrometer User's Group Newsletter No. 15, April 1975)--therefore not in alphabetical order.

(R) Registered trade names are given only as an aid to compound recognition, and do not indicate the source of the compound.